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Tertiary effluent treatment using membranes and adsorption tecnologies for industrial reuse

MEMÒRIA

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Abstract

The feasibility and reliability of membrane and adsorption technologies to reuse reclaimed water in industry applications was assessed, also the adsorbent was assessed from the point of view of micropollutants removal. To evaluate these technologies a pilot plant with a capacity of 4,0 m^3/h was constructed and operated during 8 months to assess the performance from the point of view of water yield, chemical and electrical consumption, the water quality obtained was compared with industrial requirements. The adsorbent, a nanostructured carbon (CNM) was operating continuously, with a water yield higher than 99% and without chemical consumption. The ceramic ultrafiltration (UF) with a filtration area of 22,3 m^2 was able to operate more than 30 days until CIP at 44,8 Lmh, with a chemical consumption of 1,6 L/m^3 for NaClO(15%), 20 mL/m^3 for $FeCl_3$(40%) and 0,25 L/m^3 for NaOH(50%). The RO unit, formed by 6 elements, worked with a 41% recovery and a drop pressured of 0,9 bar and a salt passage between 0,9-1,3 %, and a power consumption of 0,905 $kW \cdot h/m^3$ permeate. The water quality obtained by the couple CNM+RO met the water requirements, demonstrating that the combination of both technologies can be used to reuse reclaimed water in industry. The electrical and chemical consumption obtained by the CNM and RO system are lower than traditional UF-RO system, and the water yield is higher. The CNM removal efficiency for micropollutans was 49$\pm$38% for triazines, 57$\pm$50% for polycyclic aromatic hydrocarbons and 34$\pm$22% for pesticides.

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1. Introduction

There is a huge amount of water in the world, but 97% is salt water and only 3 % is freshwater. Due to pollutants and climate change, water scarcity and droughts are frequently. So freshwater scarcity has become a major concern in many arid and semi-arid countries worldwide (Fritzmann et al. 2007). In Europe, one third of countries are considered to be affected by water scarcity (European Commission 2012). The possibility of recycling water represents a real alternative to reduce the use of freshwater and therefore the water scarcity(Bixio et al. 2008).

Water recycling is used synonymously with water reclamation and water reuse, and is defined by the Environmental Protect Agency (EPA) as "Water recycling is reusing treated wastewater for beneficial purposes such as agricultural and landscape irrigation, industrial processes, toilet flushing and replenishing a groundwater basin".

Water reuse reduces cost and consumption of resources. One of the most important aspects is that can be treated depending on the final use, it allows more efficiency process that save resources. From an environmental point of view, it is good for ecosystem because avoid the use of water from it, not disturbing the environment. There are other advantages as reducing pollution and saving energy and cost. Reuse water can have nutrients, so in irrigation is reduced the use of fertilizers. Distribution cost is reduced because reuse use to be local, so pumping and distribution cost are reduced, these cost are bigger than reusing costs (California Energy Commission 2005).

Reuse water is gaining ground in USA, Australia, Japan and Israel. In Europe 1 billion cubic meters are reused annually, it is only the 2,4% of the treated urban wastewater. The estimated capacity of reuse water in Europe is 6 billion cubic meters. Spain reuses between 5 and 12 %, other countries as Cyprus and Malta reuse between 60 and 90 %. So there is a long way to go (European Commission 2016). The estimation of water reuse in Europe is 1.540-4000 Mm^3/y in 2025 (Hochstrat et al. 2006).

In Europe the total water for industrial purposes is 34.194 Mm3/y, which is the 18% of consumptive uses and 32% of water abstractions (EWA, 2007). Nevertheless, the water reuse in industries is low. Therefore, there is a long way to do in industrial water reuse. There are different case of reuse in industrial parks in China and Australia.

The WWTP in the industrial park of Tai Lake Basin (China) reuse 89% of industrial wastewater, so the water consume is reduced considerably. One of the most important aspects is that reuse water does not need the same requirements that drinking water, so a less amount of resources are used. The reuse process in Tai Lake Basin WWTP consist in a continuous membrane filtration, two steps reverse osmosis and electrodeionization process (Tong et al. 2013).

At Sydney Olympic Park in Australia there is a water reuse system that provides water to a population of 7.000 people. Water is used for recreation facilities, hotels, commercials premises and some industrial uses. The treatment consists in a sequencing batch reactor followed by UV. The advanced treatment uses MF and RO. Also in Australia, the Illawarra Waste Water Strategy uses a MF and RO advanced treatment to reuse water in steel industry (Wintgens et al. 2005).

An important aspect in water reuse is to comply with guidelines and laws. In Spain is regulated by Royal Decree (RD) 1620/2007. In some cases reuse water is used indirectly to produce drinking water, therefore quality aspects must be meeting. This is water for aquifer recharge and to avoid saline intrusion. Reuse water has different uses as: agriculture, landscape, cooling water for power plants and industrial uses between others (EPA 2016).

El Baix Llobregat wastewater reclamation plant (WWRP) from Barcelona (Spain) is capable of producing 50 Mm^3/y of reclaimed wastewater. The treatment of El Baix Llobregat WWRP consists in a pretreatment section, primary classification, a secondary treatment with nutrients removal and a secondary sedimentation. Next, the tertiary treatment consist in a lamellar settler with sand and anionic polyelectrolyte addition, a microfiltration, a UV light disinfection and final post chlorination with sodium hypochlorite (Cazurra 2008). The reclaimed wastewater can be used to increase the ecological flow in the lower part of the Llobregat River, to irrigate crops, to supply water to wetlands in the river deltaic areas as well as to recharge the Llobregat delta aquifer to prevent saline intrusion. But cannot be use for other applications like cooling and heating. So an advanced treatment is required to reuse the water in some industries (Bixio et al. 2006).

The aim of this project is the treatment of the Baix Llobregat WWTP tertiary effluent (“reclaimed water”) to reuse it directly in different industries. This work has been carried out in a pilot plant in the Baix Llobregat WWTP. The pilot plant has a silex-anthracite filter as pre-treatment, after this there are two treatment units or steps, a ceramic ultrafiltration (UF) and an adsorption bed with nanostructured carbon (CNM). These equipments can interchange their positions or be bypassed to study different schemes. In all the schemes the last step is a reverse osmosis (RO) unit. Water qualities for industry requires a low ionic conductivity, so in most applications is needed a RO system. Therefore, one of the objectives of the project is studying different pretreatment to reduce RO fouling and reduce chemical consumption in cleanings, meeting water requirements with the highest efficiency. In **Figure *1*** can be seen the framework of the project.

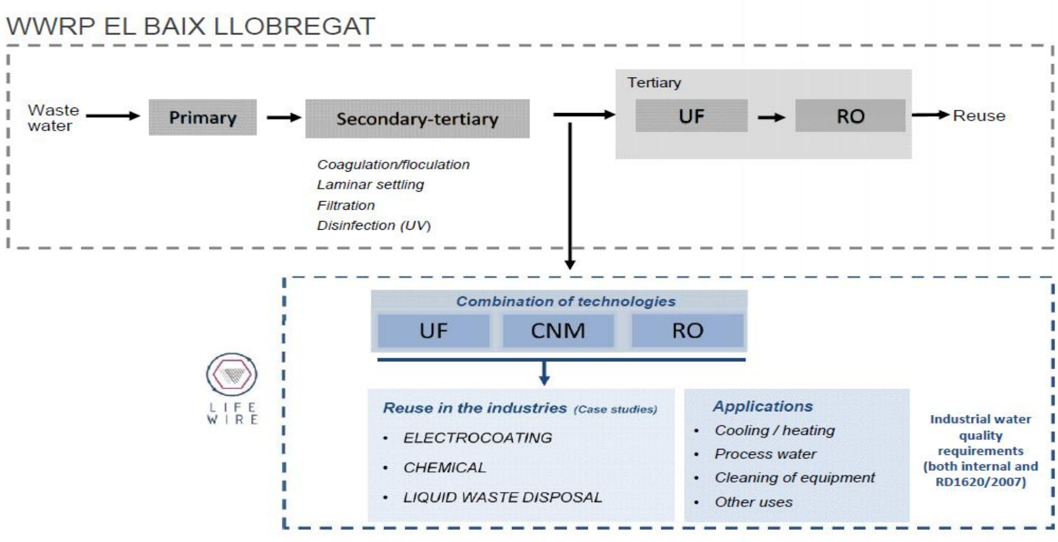

Figure 1: Framework of the water reuse treatment trains of Life Wire Project

This work is part of LIFE WIRE project (http://www.life-wire.eu/). The overall objective of LIFE WIRE is to boost industrial water reuse by making available non-conventional water resources through the reuse of urban wastewater in industries. To increase water reuse there are different instruments. In Europe has been created LIFE, it is the EU's financial instrument supporting environmental, nature conservation and climate action projects throughout the EU. Since 1992, LIFE has co-financed some 4306 projects. For the 2014-2010 funding period, LIFE will contribute approximately €3.4 billion to the protection of the environment and climate (Environmental Life Programme 2016).

The membranes are well-known technologies in advanced water treatments. RO is a key process in desalination and water reuse installation. To meet the strict requirements in legislation and the water quality requires in water reuse is usually needed a RO system. It removes the salinity from water, obtaining high-quality water. Actually the biggest problem is OPEX costs, due to the pumping energy consumption. During the lasts years has been developed two stages systems that reduces OPEX and improves water quality. This system increase CAPEX but is compensated (García and Casañas 2010). The other main cost in RO system is chemicals used to clean systems and reduce fouling. So the quality of the RO feed is very important, and therefore the pre-treatment. There are different pre-treatments needed before the RO unit. The RO feed can contain a high amount of suspended solids, colloidal material, organic material and other pollutants. These compounds cause fouling in the RO systems, so to minimize these fouling is necessary an adequate pre-treatment(Zhang et al. 2016).

UF is widely used as a RO pre-treatment because reduce turbidity, SDI_{15} and provide a constant water quality. Membranes are usually made from polymers, but have been developed ceramic membranes recently. Ceramic membranes have a higher thermal and chemical stability, pressure resistance and long lifetime than polymeric membranes, so the use of ceramic membranes is increasing (Almandoz et al. 2015) (Xu et al. 2010). Removal efficiency of ceramic membranes is high neutral and negative compounds, but low for positive compound. In mild conditions ceramic and polymeric membranes have similar efficiency, but in hardest conditions ceramics have a higher efficiency(Fujioka et al. 2014).

Inline coagulation-ultrafiltration improves UF system as RO pre-treatment. Different studies have demonstrated that it has a lower cost, footprint and energy consumption (Alizadeh et al. 2014). There are three main coagulants; polyaluminum chloride (PACl), aluminum chlorohydrate (ACH) and ferric chloride ($FeCl_3$). It has been studied that $FeCl_3$ is the most efficiency coagulant for inline coagulation with a 55% of DOC removal and 99% phosphate removal for different water qualities (Ho et al. 2015). UF pores are bigger than dissolved organic carbon (DOC) and transparent exopolymer particles (TEP) particles, so do not remove all these compounds, which are a nutrient source and a cause of biofouling. Granular activated carbon (GAC) removes DOC and TEP, 70% and 90% respectively. This improves UF permeability reducing cake deposit. The combination of GAC and UF enhance RO water obtained and reduce chemical cleanings, therefore reduce environmental impact (Monnot et al. 2016).

GAC should be combined with other technologies to be a good pre-treatment to RO system. CNM has a better properties than GAC for water treatment, so it will be assessed like a RO pre-treatment. Nanostructured materials are gaining importance in different applications like solar cell(Ali et al. 2016), analysis of pharmaceutical(Rahi et al. 2016) and diagnoses of diseases(Wang et al. 2016) between others. Recently, Blücher, a german company, has developed a nanostructured carbon. It has a larger specific surface than common activated carbon. A higher specific surface allows a higher removal capacity. Adsorption onto GAC is used in advanced WWTP to remove organic micro-pollutants and other organic substances. It is a good option because represents energy and space saving option, but it cannot be used in high dissolved organic carbon and suspended solids concentrations; this results in frequent backwash and low efficiency (Altmann et al. 2016)(Zietzschmann, Stützer y Jekel 2016).

Nowadays, there are a new concern with emerging micropollutans, those not affect the industrial water but has a big importance in drinking water. Therefore, are needed new tertiary advanced treatment to remove micropollutans, defined as pharmaceutical, personal care products, pesticides, hormones and other compounds. A conventional treatment does not remove micropollutants, and adsorption on activated carbon is presented as a low cost and efficiency alternative. Therefore, large scale experiments should be made to assess the feasibility of this technology (Mailler et al. 2016).

1.1. Definition of the case study

The water used is from tertiary effluent of Baix Llobregat WWTP in Barcelona (Spain), as can be seen in ***Figure 2***.

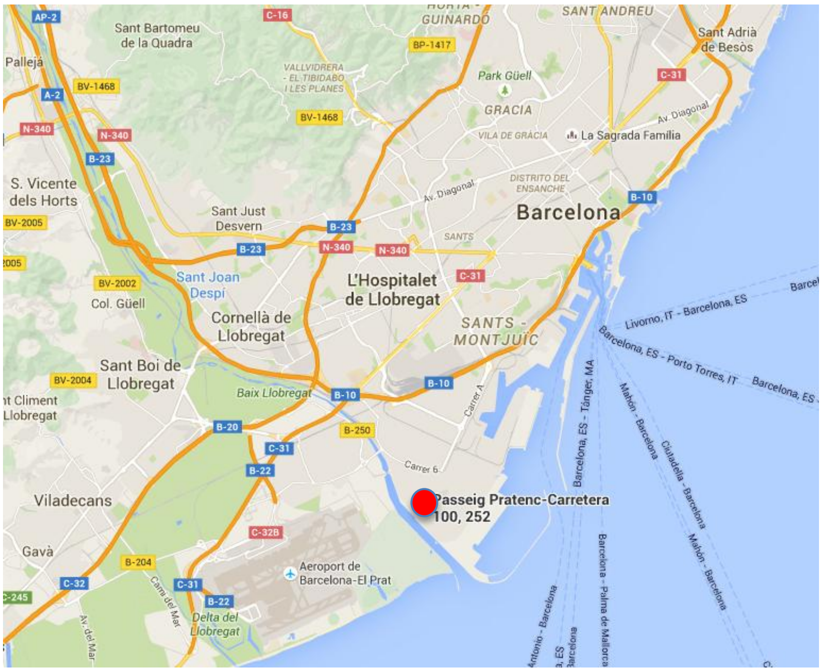

Figure 2: Location of the Baix Llobregat WWTP (Google Maps)

The main characteristics are listed in **Table 1**.Water quality is enough to some applications like irrigation but it is not enough to cooling and heating units. There are some parameters that can cause damage in these units, so those must be removed to avoid these problems.

Table 1: Characteristics of raw water of the Baix Llobregat WWTP

Parameter	Value
Iron ($mg \cdot L^{-1}$)	$0{,}041 \pm 0{,}091$
Copper ($mg \cdot L^{-1}$)	<0,002
Total hardness (ºF)	$48{,}3 \pm 3{,}5$
pH	$7{,}7 \pm 0{,}2$
Oily matter ($mg \cdot L^{-1}$)	$0{,}8 \pm 0{,}5$
Alkalinity ($meq \cdot L^{-1}$)	$2{,}9 \pm 0{,}2$
SiO_2 ($mg \cdot L^{-1}$)	$9{,}5 \pm 0{,}9$
Phosphates ($mg \cdot L^{-1}$)	$5{,}5 \pm 1{,}9$
Suspended solids ($mg \cdot L^{-1}$)	<3
Conductivity	2103 ± 111
Chloride ($mg \cdot L^{-1}$)	375 ± 75
COD ($mg \cdot L^{-1}$)	$22{,}0 \pm 3{,}5$
Bacteria ($CFU \cdot mL^{-1}$)	100 ± 44

Water quality requirements for boilers are taken from APAVE (Association of electrical and steam unit owners) and ABMA (American Boiler Manufacturers Association). Water requirements for cooling water are taken from Lenntech (Lenntech, 2016). Those requirements are shown in **Table 2**.

Table 2: Water quality requirements for boiler and cooling water (Lenntech, 2016)

Parameter	Boiler water	Cooling water
Iron ($mg \cdot L^{-1}$)	0,02	-
Copper ($mg \cdot L^{-1}$)	0,01	-
Total hardness (ºF)	0,10	14,3
pH	>8,5	7,8
Oily matter ($mg \cdot L^{-1}$)	0,05	-
Alkalinity ($meq \cdot L^{-1}$)	5	-
SiO_2 ($mg \cdot L^{-1}$)	5	-
Phosphates ($mg \cdot L^{-1}$)	20	
Suspended solids ($mg \cdot L^{-1}$)	-	0
Conductivity ($\mu S \cdot cm^{-1}$)	-	50-600
Chloride ($mg \cdot L^{-1}$)	-	250
COD ($mg \cdot L^{-1}$)	-	40
Bacteria ($CFU \cdot mL^{-1}$)	-	1000

Operation time in LIFE WIRE project is 18 months; in this time, 4 different schemes were defined to be assessed as described in **Figure *3***.

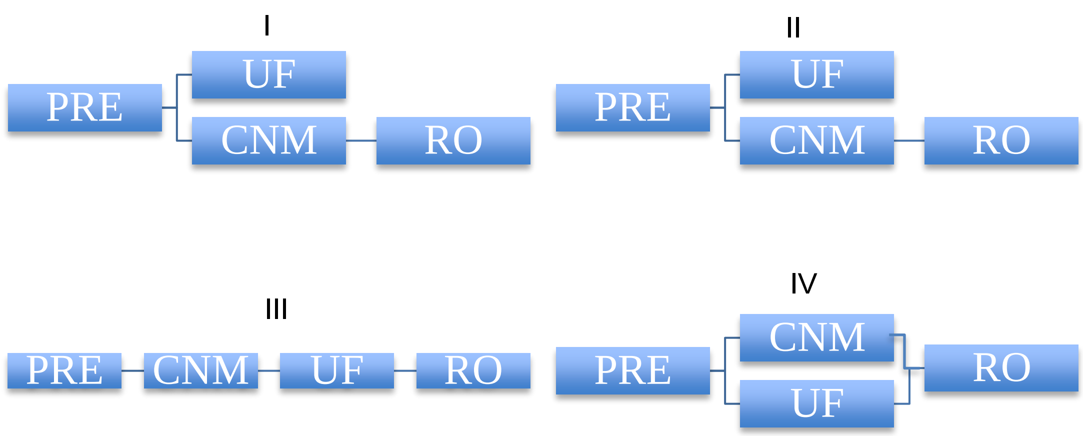

Figure 3: Water treatment schemes (I,II,III and IV) of LIFE WIRE project

In **Figure *4*** can be seen the timesheet of the project, between red bars is the time of this work. So only the configuration I will be shown in this work. The main objective of the project is to study different treatment trains as CNM, UF and RO units to meet water quality requirements for industrial applications with the highest efficiency. To obtain the highest efficiency different operation conditions will be assessed to optimize the cleaning frequency, therefore the chemical consumption. The pilot plant corresponds to a potential future satellite treatment that would be implemented in industries to meet water requirements.

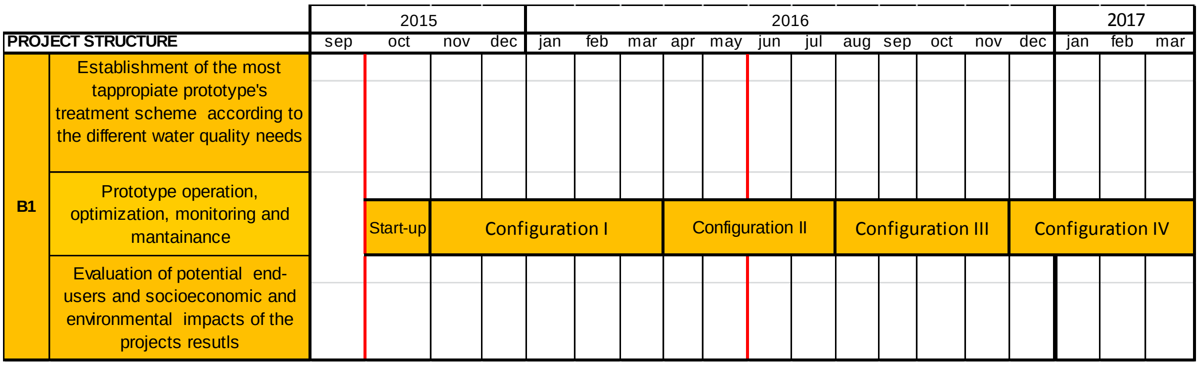

Figure 4: Schedule of the validation of the treatment schemes and TFM schedule

Therefore, the main objective of this work is to assess the configuration I, which use the CNM unit as pre-treatment of the RO, and in parallel UF. The water quality will be compared with the requirements. In this work, also the CNM will be assessed and compared with other kinds of activated carbon, and will be compare with other conventional pre-treatment for RO units, from the point of view of energy and chemical consumption. In parallel, the CNM removal efficiency will be assess from the point of view of micropollutants. The ceramic UF operation will be assessed and compared with polymeric UF in tertiary treatments.

2. Theoretical background

2.1. Adsorption on nanostructured carbon

Nanostructured carbon is considered as activated carbon. The specific surface area of nanostructured carbon is higher than activated carbon, enhancing removal efficiency. In adsorption process, particles (adsorbate) in water (bulk phase) are attached into carbon surface (adsorbent).

Adsorption can be reversible or irreversible. Reversible adsorption is when union forces are weakly as van der Waals forces, when a compound is adsorbed with this forces can be easily removed by a backwash. Irreversible adsorption occurs when chemical bonds are formed, so it is difficult to remove it.

Carbon granules have different pore sizes as it is shown in **Figure 5**; macropores (diameter >50 nm), mesopores (diameter 2-50 nm) and micropores (diameter >2 nm). Suspended solids with diameters higher than 50 nm are attached in carbon surface and can be easy removed.

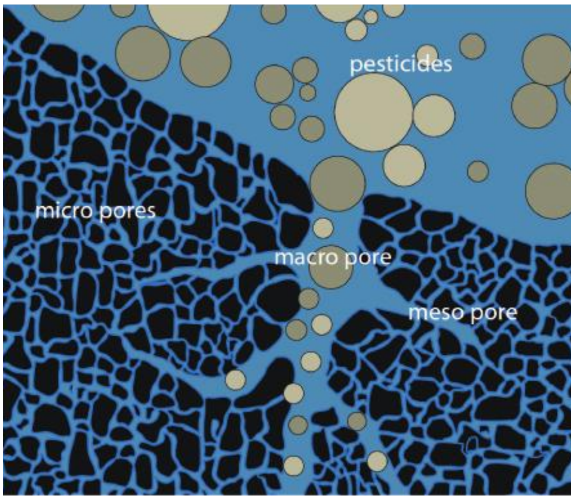

Figure 5: Pore types in carbon particles (De Moel et al., 2014).

Smaller compounds are adsorbed in mesopores and micropores and are hardly removed from carbon pores. During filtration time pollutants are adsorbed in pores, when macropores are full, inlet pressure in column is high and is necessary a backwash. Between cycles, flow is reduced due to higher bed resistant. After a backwash the major part of compounds are removed from carbon but some particles are strongly attached to internal surface and cannot be removed. Along the operation time more particles are retained in carbon, after some cycles carbon is saturated and it is necessary the carbon regeneration. When carbon is saturated, it is in equilibrium and adsorption does not occur. During thermal carbon regeneration there are a 4-8% lost of carbon due to carbon oxidation (De Moel et al., 2014).

Carbon regeneration depends of the removal efficiency that is required by the objective target. If a high quality is needed, the regeneration time will be shorter than when a low quality is needed. In this work the objective is obtain the removal curve, therefore there is not a quality target and the carbon will operate until removal efficiency is near zero.

2.2. Ultrafiltration

Nowadays, membranes are used in water treatment to remove compounds or particulate matter. Microfiltration and ultrafiltration are used to remove colloidal substances and microorganism. Nanofiltration and reverse osmosis also remove dissolved species, as neutral and ionic species. So previously a RO systems is necessary a MF or UF. In this case ceramic ultrafiltration is used. MF removes bacteria and larger viruses, but UF also removes smaller viruses due to the smaller pore size. A description of the range of operation is shown in **Figure 6**.

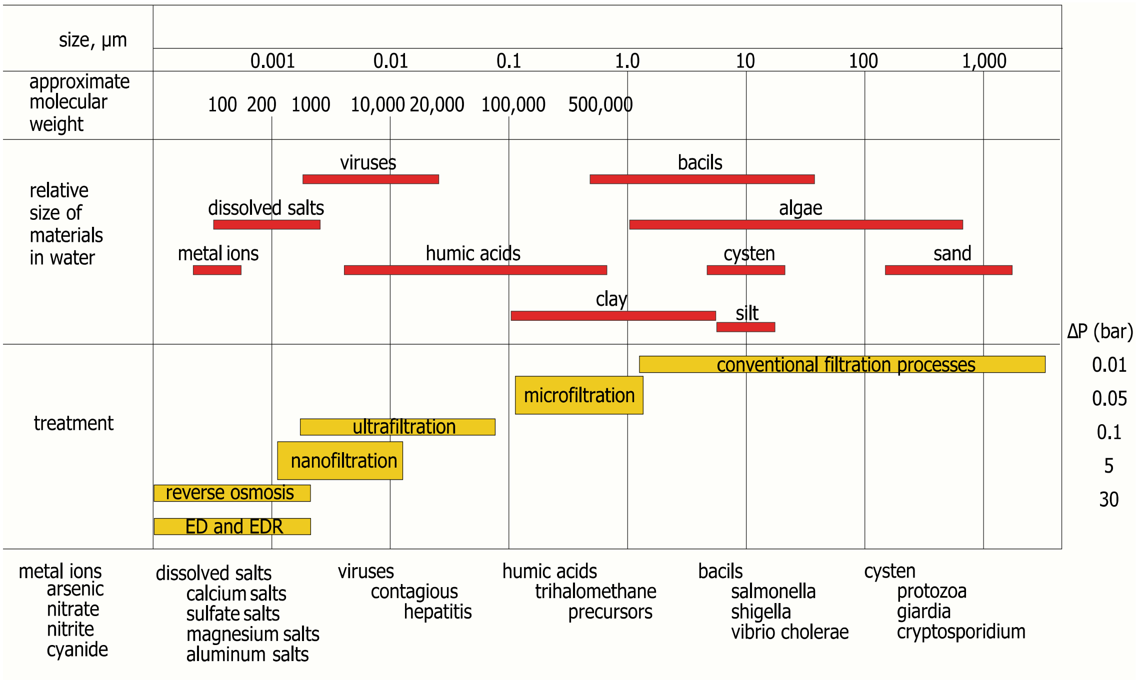

Figure 6: Overview of different filtration processes and sizes of compounds removes (De Moel et al., 2014).

The compounds that are removed can be measured by their size (μm) or the molecular weight cut-off (MWCO). MWCO is the molecular weight of spherical particles that have a

90% of reject. This is measured in Dalton; UF removes in the range of 10 to 500 kD. The objective of UF as a RO pre-treatment is to remove suspended solids, heavy metals, bacteria and viruses.

To produce a high amount of water is needed a large filtration area. To obtain this area, membranes are manufactured in modules where is possible to obtain the largest membrane area in the smallest volume. This surface is called specific surface, in a membrane is defined as:

$$A_{spec} = \frac{A_{mem}}{V_{module}} = \frac{n \cdot \pi \cdot d \cdot L}{\pi \cdot D^2/4} \tag{1}$$

UF membranes are semi-permeable, so the smaller particles can pass thought it and the bigger compounds are rejected. In a membrane system there are two outlet flows; the water that pass through the pores is called permeate and the particles that are rejected are called concentrate. In this UF system there is not concentrate flow, all the water out as permeate and the particles are retained in membrane walls, this is called dead-end filtration mode. After filtration time a backwash is made and pollutants are remove from membrane surface, this is a mechanical cleaning, the filtration time plus backwash time is called filtration time or filtration cycle.

Membranes can operate in two modes; the water flux decreases and the filtration pressure is constant or the pressure increases and the flux is constant. In this case flux is constant because it can be the feed of the RO system and must guaranteed a constant flux.

The mass balance for a filtration cycle of the UF system is:

$$Q_f = Q_p + Q_{BW} \tag{2}$$

The recovery is the water free of compounds obtained divided by the water that is treated, so:

$$\gamma(\%) = \frac{V_p - V_{BW}}{V_f} \cdot 100 \tag{3}$$

To obtain a high recovery is needed a long filtration time and reduce the backwash time. Usually backwash flow is two times filtration flow.

During ultrafiltration operation transmembrane pressure (TMP) is an indicator of filtration process. Flux and TMP are related by:

$$J = \frac{Q}{A_{memb}} = \frac{TMP}{\nu \cdot R_{tot}} \quad (4)$$

Where,

J is flux [$m^3/(m^2 \cdot h)$]

Q is volume flow [m^3/h]

A_{memb} is membrane surface area [m^2]

TMP is transmembrane pressure [Pa]

ν is dynamic viscosity [Pa/s]

R_{tot} is total resistance [m]

The permeability of the membrane depends on temperature, so the TMP depends on temperature. At high temperatures flux grow up to 3% per ºC. Therefore, it is necessary normalized the flux to study the membrane fouling. TMP is defined as the differences between permeate and feed pressure. The hydraulic pressure drop is small, and can be known at the beginning of the operation, it is measured when the membrane is new and with demineralized water. So the fouling is study through the TMP during operation. During filtration compounds are suspended in the membrane pores and a cake layer is formed, increasing the resistance and so TMP, this effect is known as fouling. The total resistance is the sum of all the kinds of fouling:

- Membrane resistance
- Pore blocking
- Adsorption in the pores
- Cake resistance
- High concentration of dissolved substances near the surface

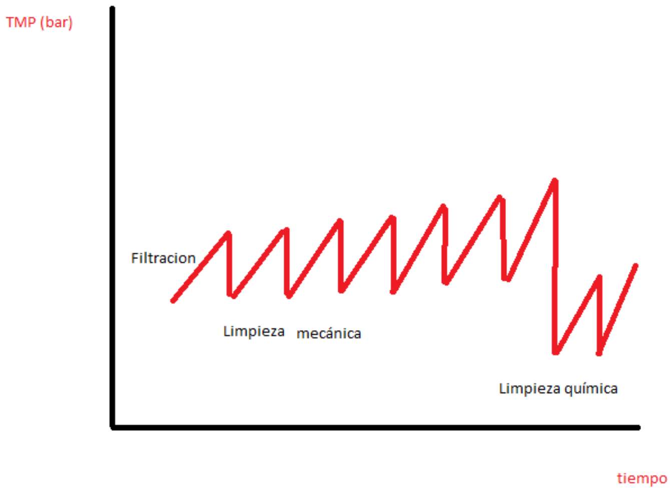

Figure 7: Evolution of TMP during filtration cycles

There are different kinds of mechanical and chemical cleanings (**Figure 7**). The objective of the mechanical cleanings is to remove the cake layer formed between filtration cycles. There are some compounds that are not remove during mechanical cleanings and the cake layer grows up. After some cycles is needed a chemical cleaning to reduce cake layer. When CIP is not enough, membranes must be replaced. One alternative to reduce fouling is using an inline-coagulation.

Coagulation consist of the destabilization of the negatively charged particles in the water through the dosing of a coagulant, so flocks are formed and can be easily remove, reducing fouling (De Moel et al., 2006). The effect depends of coagulation doses. Water with a low amount of organic matter needs a lower consumption of coagulant, so inline coagulation is the best alternative. Coagulation reduces pore blocking, higher permeability and reduces strength of adhesion of particles to the membrane surface(Alizadeh et al. 2014). To mix the coagulant with water it is necessary a mixer, in this case Raschig rings are used. The mixer increase contact time to allow flocks formation.

2.3. Reverse osmosis

Osmosis is the process by which the two layers of a semi-permeable membrane tend to equalize their salt concentration. When a pressure is applied on the higher concentration side, water flows thought the membrane but salts are retained and water free of salts is obtained, this process is known as reverse osmosis.

RO also needs a high specific surface; to obtain it spiral-wound membranes are used

(**Figure 8**). Those have a large specific surface, around 1000 m^2/m^3. In spiral-wound membranes water is fed via feed spacers, these are layers between membrane sheets. An element is a number of membrane sheets twisted around a central permeate collecting tube. To reduce cost, different elements are put in one membrane module because a high-pressure vessel is necessary and it is expensive.

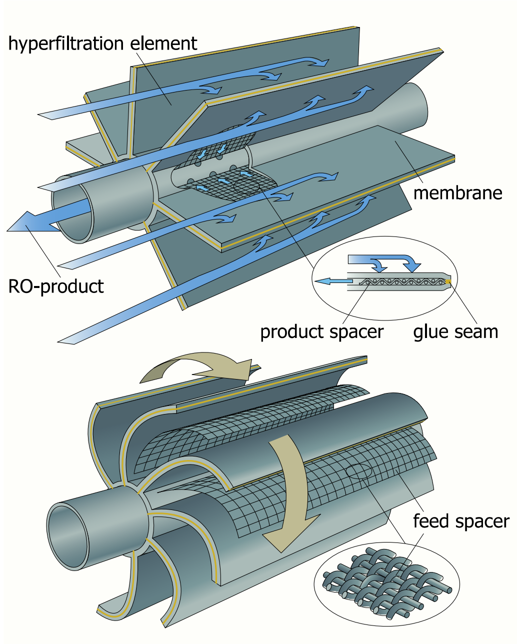

Figure 8: Principle of spiral-wound membranes (De Moel et al., 2014).

In osmosis process there are three different flows; feed, permeate and reject. Salt concentration in reject is higher than permeate concentration. Therefore water mass balance is:

$$Q_f = Q_r + Q_p \tag{5}$$

So recovery is defined as:

$$\gamma(\%) = \frac{V_p}{V_f + V_{flushing}} \tag{6}$$

Recovery varies between 40 and 95% depends of water salt content. When higher is the recovery higher is the fouling and scaling, so there must be a balance to obtain the highest efficiency.

Membrane permeability depends of temperature, feedwater composition, feed pressure and recovery. To study the fouling and scaling flow must be normalized, because a decrease in permeate flow could be due to a temperature change. Thanks to normalization can be known if changes in permeability are from fouling or not. It is estimated that a temperature drop of 4°C cause a permeate flow decrease of 10 % (De Moel et al., 2006).

DOW has made a spreadsheet that normalizes flow and salt pass. It is a very helpful tool because facilitates the calculation (DOW 2009).

$$Q_s = \frac{P_{fs} - \frac{\Delta P_s}{2} - P_{ps} - \pi_{fcs}}{P_{fo} - \frac{\Delta P_o}{2} - P_{po} - \pi_{fco}} \cdot \frac{TCF_s}{TCF_0} \cdot Q_o \tag{7}$$

Where,

P_f is feed pressure [bar]

$\Delta P/2$ is one half-device pressure drop [bar]

P_p is product pressure [bar]

Π_{fc} is osmotic pressure of the feed-concentrate mixture [bar]

TCF is temperature correction factor

Q product flow [m^3/h]

Subscript s represents standard condition at the beginning of the operation

Subscript o represents operating condition

Temperature correction factor is calculated as

If T ≥ 25°C

$$TCF = EXP\left[2640 \cdot \left(\frac{1}{298} - \frac{1}{273+T}\right)\right] \tag{8}$$

If T ≤ 25°C

$$TCF = EXP\left[3020 \cdot \left(\frac{1}{298} - \frac{1}{273+T}\right)\right] \tag{8}$$

Osmotic pressure for concentration lower than 20.000 mg/L is calculated as

$$\pi_{fc} = \frac{C_{fc} \cdot (T + 320)}{491000} \tag{9}$$

C_{fc} is approximated for

$$C_{fc} = C_f \cdot \frac{\ln\left(\frac{1}{1-\gamma}\right)}{\gamma} \tag{10}$$

Finally, the normalized permeate TDS is calculated from

$$C_{ps} = C_{po} \cdot \frac{P_{fo} - \frac{\Delta P_o}{2} - P_{po} - \pi_{fco} + \pi_{po}}{P_{fs} - \frac{\Delta P_s}{2} - P_{ps} - \pi_{fcs} + \pi_{ps}} \cdot \frac{C_{fcs}}{C_{fco}} \tag{11}$$

RO is used to remove dissolved species as salts and micropollutants. To prevent fouling is needed an adequate pretreatment, there are conventional pretreatment as coagulation, flocculation, sedimentation or filtration. In this case new technologies are used as pretreatment (CNM and UF). Nevertheless fouling occurs due to pollutants removed, salt content in membranes form salt precipitated, this process is called scaling.

To reduce scaling some chemical products are used, mainly acids. This anti-scalants removes seeding material and help to not excess the solubility product. Scaling occurs when saturation index (SI) is exceeded. To prevent it cross-flow velocity can be increased but this needs a high-energy consumption, so use of anti-scalants is preferred. Also to reduce fouling and scaling is used flushing, it consist is pass osmotic water in the filtration flow direction to remove some pollutants.

3. Materials and methods

3.1. Pilot plant

Pilot plant of LIFE WIRE project is a modular plant that consists in four units; the main characteristics of units are summarized in **Table 3**. The prototype has been operating continuously since October 2015. The raw water comes from El Baix Llobregat WWRP; the quality is enough to meet the Spanish Royal Decree 1620/2007 requirements, but not enough to other applications, therefore is needed an additional treatment. Pipes and Instrument Diagram is show in **Annex A**.

Table 3: Summary of characteristics of the equipment

	PRE	UF	CNM	RO
Type	Dual filtration media: anthracite-silex	Ceramic membrane-**Likuid L91**	Nanostructured carbon - **SARATECH**	Spiral wound 4 inches membranes HYDRANAUTCS: **LFC3-LD-4040**
Configuration	Grabel: 40 kg Silex: 80kg Anthracite: 75 kg	1 module of 91 membranes: -$D_{channel}$: 3,5 mm -Channels: 19 -L: 1178 mm -Pore size: 100 nm -Total filtration area: 22,3 m^2	One filter with 160 kg of adsorbent: -H:1250 mm -D:500 mm	6 membranes in 2 stages: -1st stage: 2 vessels of 2 membranes each -2nd: 1 vessel with 2 membranes

In **Table 4** are shown the technical data of CNM. In **Figure 9** is shown a picture of CNM, can be seen that has a spherical shape, other kinds of activated carbon has pellet shape.

Figure 9: Nanostructured carbon material, picture supplied by the manufacturer

Table 4: Technical data of Saratech adsorbent (CNM) supplied by the manufacturer

Technical data of SARATECH® spherical high-performance adsorbents

physical properties	test method	value	dimension
BET-surface	ASTM-6556-04	600 – 2.100	m^2/g
ball pan hardness	ASTM D3802-79/05	99,5	%
ash content	ASTM D2866-94/04	0,1	%
tap density	ASTM B527-93/00	250 - 800	kg/m^3
bulk density	ASTM B311 B328	250 - 800	kg/m^3
water content	ASTM D2867-04	‹ 1	%
abrasion strength	AWWA B604-96	› 99	%
particle size	internal method CAMSIZER	0,01 – 0,7	mm

These units operate individually allow different schemes. All the units have storage tanks that permit more flexibility during operation time. A sketch of the prototype units can be seen in **Figure 10**.

The first step of the process is the pretreatment, it consist in a dual media filter (silex, anthracite, 4,0 m^3/h). After it there are two possible ways, UF (ceramic membrane Likuid L91, filtration area 22,3 m^2, 2,0 m^3/h) or CNM. CNM is a nanostructured carbon based material manufacturer by Blücher (116 kg of Saratech adsorbent, 2,2 m^3/h nominal capacity). The final step is a RO unit (Hydranautics LFC3-LD-4040, 2,1 m^3/h nominal capacity).

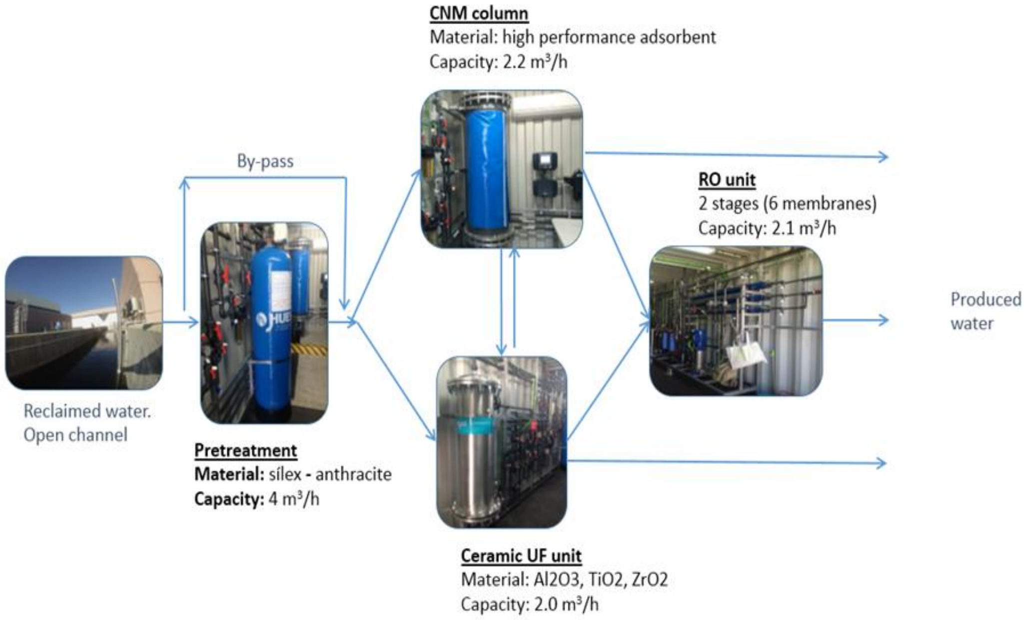

Figure 10: Sketch of units and main characteristics

The CNM unit can work continuously with a feed flow of 2,2 m^3/h, the feed pressure was monitored continuously. When inlet pressure reaches a value of 1,5 bar (established by the manufacturer) a BW is needed. BW consist in filtering, from the bottom of the column to the top, osmotised water at 0,2-0,6 m^3/h during 15 min, 3 times, to dislodge the foulants accumulated on the system.

The ceramic UF worked with a constant flow of 1,0 m^3/h, increasing the TMP during filtration time. After the filtration time a BW is needed. BW consist in pass osmotised water through the membrane with a flow of 4,0 m^3/h and pressure of 1,8 bar. The BW protocol is in three steps. In the first step (backwash), water is fed by permeate side and goes out by the purge side, this step allows separate the fouling from the membrane. In second step (backwash + discharge), water is fed also by permeate side and goes out by the bottom of the module. In the last step (discharge), the procedure is the same as in second step, but air enters by purge side to cause turbulence and remove the foulants, which are more strongly attached to the membrane. UF CIP consist in clean the membrane with chemicals, the first step consist in rinsing the membrane with 2,00 m^3/h of osmotised water during 10 minutes to remove the particles that are weakly attached to the membrane surface. The second step is the chemical cleaning with a basic solution, 1% of sodium hypochlorite and 0,5% of sodium hydroxide, first during 20 minutes at ambient temperature and after at 30^{o}C. The membrane is rinsed with water again, and an acid cleaning with nitric acid at 1% is made at 30^{o}C. During the chemical cleanings at 30^{o}C the TMP is monitored and the cleaning lasts until that the TMP is not reduced more. Finally the membrane is rinsing again. All the steps are made with a flux of 2,00 m^3/h.

The RO consist in a 2-stage system. Two vessels in parallel form the first stage, the reject of the first stage is the feed of the second stage, which is formed by one vessel. All the vessels contain two elements, so there are six elements in total. To control the system performance, flow meters and conductivity are put in all the currents. Flushing operations consist in filter osmotised water with a flow of 2,0 m^3/h during 90 seconds. To prevent membrane damage and decrease the risk of scaling, a reducer (Genesys RED) and an antiscalant (Genesys LF) are dosed. Redox is monitored on line to activate the reducer dose.

PRE and CNM have in the inlet and in the outlet filters to avoid the input and output of filtration media and carbon. UF has in the inlet one filter because the feed water can be from PRE or CNM and can contain big particles. RO is more delicate than the other units; so one microfilter is put in the inlet.

3.2. Monitored parameters

Some parameters are measured online to control the operation and know if the units are operating correctly. Those parameters, that can be seen in **Table 5**, are measured each 2 seconds but to work with a low amount of date, graphics are made with a frequency of take date of 30 seconds.

Table 5: Parameters monitored online

Unit	Parameter	Current
PRE	Pressure	In/out
	Flow	In
	Turbidity	In
UF	Pressure	In/out
	Turbidity	In/out
	Flow	Out
	Temperature	In
CNM	Flow	In
	Pressure	In/out
	Turbidity	Out
RO	Conductivity	In/Reject/Permeate
	Flow	In/Reject/Permeate
	Pressure	In/Reject/Permeate
	Temperature	In/Permeate
	Redox	In
	pH	In

The methods used to measure the different parameters were; temperature by resistivity (IFM TA3130), conductivity by electrometry (Hach Lange PH28P), redox by potentiometry (Hach Lange RX38P), pH by potentiometry (Hach Lange PH28P) and turbidity by nephelometry (Hach Lange 1720E).

Pretreatment and CNM are simply filtration/adsorption process, so to control the operation is only needed to monitor pressure and flow. If there were fouling in the unit, it would be known easy through the studying of the flow and pressure decrease.

Membrane processes are more complex and more parameters must be monitored. In UF operation, PID controller controls outlet flow rate and it is constant during operation time, so the transmembrane pressure is increased and fouling are known through this parameter. Membrane permeability changes with temperature, so this parameter must be monitored to calculate normalized transmembrane pressure and know UF fouling.

To calculate normalized parameters in RO unit, it is needed the same parameters that in UF and also conductivities. Redox indicates if there is chlorine in RO feed, this compound cause damage in RO membrane and must be removed by the adding of sodium metabisulfite (Genesys Red). When redox is higher than 200 mV, the dosage of Genesys Red is activated and redox is reduced. If redox is higher than 600 mV the water contain chlorine and RO unit is stopped to avoid damage in the membrane. The pH is monitored to protect the unit, if there is a big variation in pH; the unit is stopped to prevent damage.

Turbidity is monitored to protect the units. The equipment's are design to operate with a low turbidity, so it must be controlled. If there were a peak in turbidity, units would be stopped to prevent damage.

3.3. Analytical methods

Physiochemical parameters are analysed weekly, microbiological monthly. Both are measured in Raw Water, PRE/UF/CNM/RO permeate. Those parameters are analysed by the laboratory of Área Metroplitana de Barcelona (AMB) in the WWTP of Gavá-Viladecans. There are near to 150 parameters, but not all of them will be study in this work. In **Table 6** are summarised the most important to fulfil the objective of this work. The analytical procedure of laboratory work is not one of the objectives of this project, so it will not be explain here. In any case, it should be mentioned that analytical procedures used by the AMB, are methods recommended by the Standard Methods of wastewater analysis.

Table 6: Methods used to analyse the selected group of parameters for monitoring water quality evaluation (AMB)

Parameter	Method	Description
Iron	ICP/MS	Acid digestion / Ion coupled plasma-Mass Spectrometry (Perkin-Elmer Nexion 300x)
Copper	ICP/MS	Acid digestion / Ion coupled plasma-Mass Spectrometry (Perkin-Elmer Nexion 300x)
Total hardness	Calculation	From Ca,Mg
pH	Electrometry	Mettler inlab powepro
Oily matter	Gravimetry	O&G-Solid-phase -extraction-gravimetry
Alkalinity	Calculation	From HCO3, CO3
SiO_2	ICP/MS	Fluorhidric digestion / Ion coupled plasma-Mass Spectrometry
Phosphates	photometry	molibdovanadate method
Conductivity	elctrometry	Mettler Inlab 731
Chloride	Ion chromatography	DIONEX DX-120 EGC-KOH (AS14)
COD	photometry	Dichromate chemical oxigen demand ultra-low range 5-40 mg/L
Total aerobic	UNE-EN-ISO 6222:1999	Counting the colonies on a nutrient agar culture medium after 48 h incubation at 36 ° C in aerobic conditions
Legionella	Enzyme immunoassay	Immunomagnetic capture and colorimetry
Pseudomonas	Pseudalert (DST)	Enzymes of the bacteria stick to the substrate splitting fluorescent molecules
Coliforms	Colilert (DST)	Enzymes of the bacteria stick to the substrate splitting colored molecules
E.Coli	Colilert (DST)	Enzymes of the bacteria stick to the substrate splitting fluorescent molecules
Enteroccoci	Enterolert (DST)	Enzymes of the bacteria stick to the substrate splitting fluorescent molecules

To study CNM removal efficiency from the point of view of micropollutants, five families of micropullutans has been analysed; triazines, benzene, toluene, ethylbenzene and xylenes (BTEX), polycyclic aromatic hydrocarbons (PAH), pesticides, polychlorinated biphenyls (PCB).

Table 7: Micropollutants assessed in CNM operation

Triazines	**PAH**	**Pesticides**	**BETEX**	**PCB**
Atrazine	Acenaphthene	a-Hexachlorocyclohexane	1,1 Dichloroethane	2,2,3,4,4,5,5-hexachloro biphenyls
Atrazine-desethyl	Acenaphthylene	Aldrin	1,1 Dichloroethane	2,2,3,4,4,5-hexachloro biphenyls
Atrazine-desisopropyl	Anthracene	Alpha-endosulfan	1,1,1 Trichloroethane	2,2,4,4,5,5-hexachloro biphenyls
Sebutilazina	Benzo (a) anthracene	Beta-endosulfan	1,1,2 Trichloroethane	2,2,4,5,5-pentachlorobiphenyl
Simazine	Benzo (a) pyrene	b-hexachlorocyclohexane	1,1,2,2 - Tetrachloroethane	2,2,5,5-tetrachlorobiphenyl
t-butilazina	Benzo (a) fluoranthene	Chlordane	1,2 Dichloroethane	2,3,4,4,5-pentachlorobiphenyl
	Benzo (a) pyrene	d-Hexachlorocyclohexane	1,2,4 Trichlorobenzene	2,4,4-trichlorophenyl
	Benzo (k) fluoranthene	Dieldrin	1,3 Dichlorbenzene	
	Chrysene	Endrin	1,4 Dichlorbenzene	
	Dibenzo (a, h) anthracene	Endrin Aldehyde	Other halogenated	
	Phenanthrene	Endrin Ketone	Bromodichloromethane	
	Fluoranthene	g-HCH (Lindane)	Bromoform	
	Fluorene	Heptachlor	cis-1,2-Dichloroethane	
	Indeno (cd) pyrene	Heptaclorepoxid	Chlorobenzenes	
	Naphthalene	Metoxichlor	Chloroform	
	Pyrene	Nonachlor	Methylene Chloride	
		p-p'-DDD	Chlorodibromomethane	
		p-p'-DDE	Perchlorethylene	
		p-p'-DDT	Carbon Tetrachloride	
			trans-1,2 -Dichloroethane	
			Trichlorethylene	

			Acetates	
			Acetone	
			Acetonitrile	
			Methyl Acrylate	
			Alkanes	
			Alkylbenzenes	
			Other non-halogenated	
			Benzene	
			Estirene	
			Ethylbenzene	
			Iso-propylbenzene	
			m+p Xylenes	
			Methyl Methacrylate	
			methyl ethyl ketone	
			Methyl isobutyl ketone	
			Methyl tert-butyl ether	
			o-Xylene	
			Dimethylsulfide	
			Terpenes	

4. Results and discussion

In configuration I (**Figure 11**), PRE flow is 4 m^3/h. UF operate with 1 m^3/h and CNM operate with a flow of 2 m^3/h. RO feed flow is also 2 m^3/h Other conditions were studied, working CNM and RO unit with 1,5 m^3/h, the objective of this conditions was studied the CIP requirements of RO, and different filtration velocities of CNM.

Figure 11: Configuration 1, operation time November 2015-May 2016

4.1.1. Ultrafiltration performance

During the UF start-up was observed a high velocity in membrane fouling, therefore the UF could not operate continuously. Different backwash times were tested to obtain the best operation conditions; the BW conditions are shown in **Table 8**. The initial conditions were 80 seconds for backwash (BW), 10 for BW and discharge (D) and 40 seconds for D, these conditions were established by the manufacturer. The second conditios tested were 50 s for BW, 20 s for BW+D and 50 s for D, the ΔTMP was reduced from 0,2 bar/h to 0,1 bar/h for 44,8 Lmh (1,00 m^3/h). Monitoring the transmembrane pressure during BW was shown that the first step (BW) was no effective and the cleaning times were very long, therefore a new BW conditions were tested, BW 0s, BW+D 20 s, and D 25 s. The new conditions reduced the ΔTMP from 0,7 bar/h to 0,5 bar/h for 89,7 Lmh (2,00 m^3/h).

Table 8: BW conditions tested in UF

Lmh ($L \cdot m^{-2} \cdot h^{-1}$)	BW (s)	BW + D (s)	D (s)	ΔTMP (bar/h)
44,8	80	10	40	0,2
44,8	50	20	50	0,1
89,7	50	20	50	0,7
89,7	0	20	25	0,5

The different BW conditions tested were not enough to operate continuously and the CIP frequency was very high. Jar Test was made to conclude that a coagulant dose could reduce the membrane fouling. The first step was the optimization of coagulant dose from the point of view of membrane fouling. **Figure 12** shows the variation of the transmembrane pressure changes with different coagulation dose and without coagulation. It can be seen that the biggest TMP increase measured for experiments was without coagulant, so the effect of the coagulant is positive.

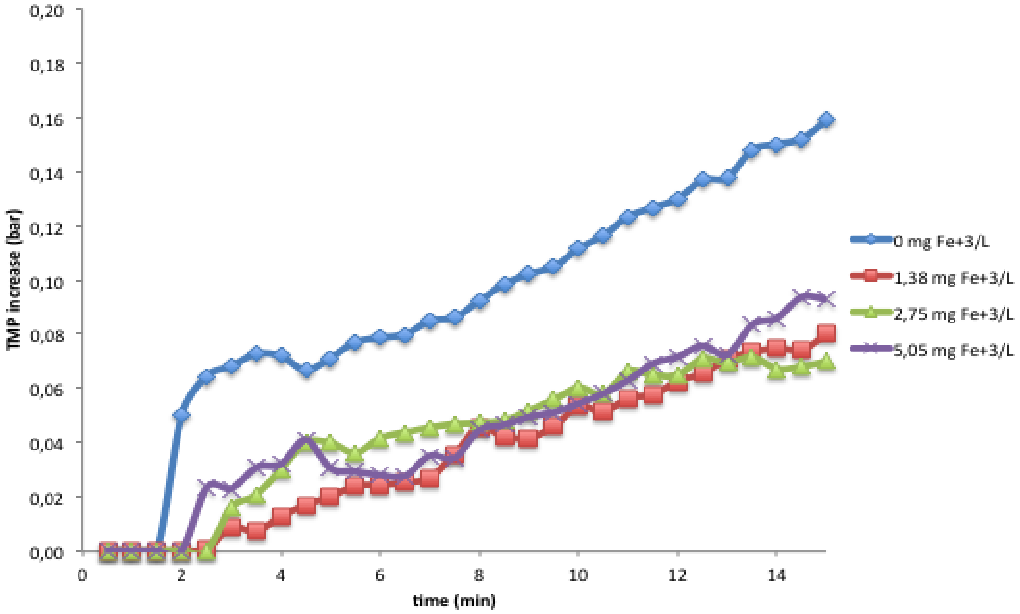

Figure 12: Variation of TMP as a function of filtration time (15 min) for different coagulant ($FeCl_3$) dose

To study the effect of the coagulant dose, three experiments were made. In each experiment UF was operating during 4 described in **Table 9**. In all the experiments the flux was 89 LMH ($L·m^{-2}·h^{-1}$).

Table 9: Effect of coagulant dose (mg Fe^{+3}/L) on ΔTMP along the filtration cycles

Coagulant dose (mg Fe^{+3}/L)	ΔTMP (bar/h)
1,4	0,28
2,8	0,25
5,1	0,35

Without coagulant TMP increases 0,50 bar/h, much higher than with coagulant. A coagulant dose of 2,8 mg Fe^{+3}/L reduces the increases of TMP to 0,25 bar/h. In **Table 9** is shown that the best coagulant dose is of 2,8 mg Fe^{+3}/L, from the point of view of membrane fouling.

Despite of coagulant dose, the CIP frequency was very high and, therefore the chemical consumption very high. During CIP the membrane is rinsed with water (forward flush), monitoring the TMP during CIP was observed that the TMP were reduced with rinsing, therefore a new BW protocol was tested, with an initial rinsing of 10 seconds to displace the particular matter from the membrane. The new BW protocol reduced the TMP from 0,08 bar/h to 0,013 bar/h for 44,8 Lmh (1,00 m^3/h). These conditions were assessed during a few days; were observed that the variability of inlet water quality caused changes in ∆TMP. Therefore these conditions were not enough to obtain a reduced chemical consumption.

At this point, the BW efficiency was not enough and the hydraulic design of the UF was assessed. In **Figure 13** is shown the UF module, can be see the top and bottom of the module, these diameters are smaller than usual diameters in UF units. Therefore the module was open and inspected. The objective was to determine if there were pollutants retained in the top and in the bottom of the module due to the small diameter of the top and bottom pipes.

Figure 13: Ultrafiltration unit

In **Figure 14** and **Figure 15** are shown the inside of the UF module is shown. In both figures can be seen that the module is free of particular matter, therefore the hydraulic design is well-done.

Figure 14: Detail of the top of the UF module

Figure 15: Detail of the bottom of the UF module

The next step was to assess the scaling in the membrane through acid chemical enhanced backwash (CEB). CEB consist in add chemicals in BW protocol. During CEB the BW times were longer than during BW without chemicals. Acid CEB were made with nitric acid at 0,5% and reduced the TMP 0,3 bar/CEB, but the TMP was destabilized after it. Basic CEB with sodium hypochlorite at 0,5% were tested, this reduced the TMP from any value of TMP to the initial TMP after CIP. Therefore scaling is not the problem of operation, and the best solution was operating with basic CEB. The BW was not effective, therefore this step was eliminated from the process and only one basic CEB each 6 hours was made.

In **Figure 16** can be seen the evolution of TMP with the permeate volume. During filtration time TMP increase and during basic CEB the TMP is completely reduced. The next step will be the optimization of CEB conditions from the point of view of frequency, concentration and time. Between 0 and 5 m^3 of treated volume, the acid CEB was made and can be seen the destabilization of TMP, therefore it was remove of the process.

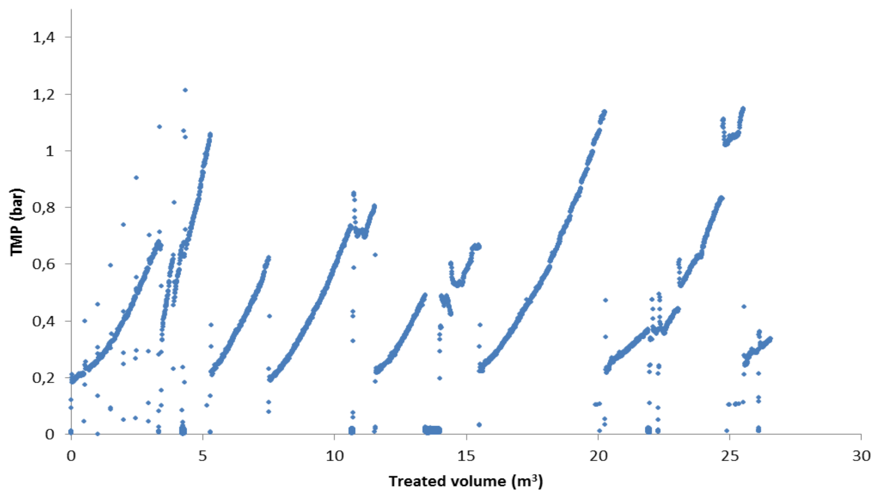

Figure 16: Evolution of TMP during UF operation as a function of treated volume

In **Figure 17** is shown the evolution of TMP during basic step of the CIP, and in **Figure 18** is shown the evolution of acid step. In both cases can be seen that reached a point the TMP is not reduced and the slope is near zero, therefore the CIP is finished. Basic step lasts near 5 hours and acid step 2 hours. The basic step reduces TMP from 2 bar to 1,1 and acid step from 1,1 to 0,3 bar. At the beginning of the operation the CIP protocol was with 60 min of each step, but was observed the curve and therefore the time was increased.

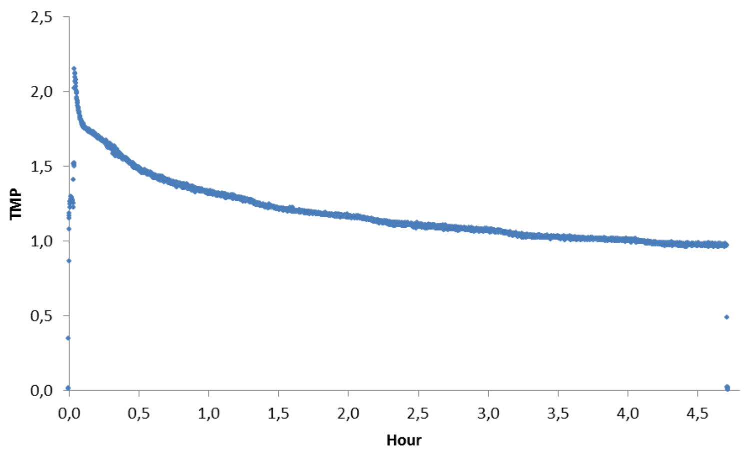

Figure 17: TMP evolution during basic stage as a function of time

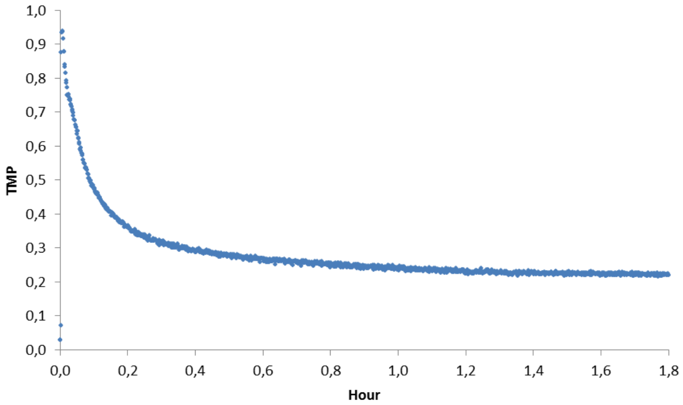

Figure 18: TMP evolution during acid stage as a function of time

To have a reference value, the results obtained in ceramic UF has been compared with the values obtained in a tertiary treatment in a WWTP in Madrid (Ordóñez et al. 2011). In this process the UF is polymeric and work between 25 and 45 Lmh, therefore the results can be compared.

Table 10: Comparison of BW, CEB and CIP frequency between work made in Madrid by Ordóñez et al. and LIFE WIRE project.

Parameter	Polymeric UF by Ordóñez et all.	Ceramic UF by LIFE WIRE
Flux (Lmh)	29	45
BW frequency (min)	65	-
Basic CEB frequency (h)	24	6
Acid CEB frequency (h)	72	-
CIP frequency (days)	12-14	>30

In **Table 10** is compared the best results obtained in Madrid and in LIFE WIRE experiments. In ceramic UF the BW was not efficiency, therefore these step in cleanings was removed. The BW frequency in polymeric UF was 65 minutes, with one basic CEB (0,015% NaClO) per day, one acid CEB (0,8% citric acid) each three days. In these conditions the CIP frequency was between 12 and 14 days. Ceramic UF operate in a stable way with one basic CEB (1,00% NaClO) each 6 hours, and without acid CEB. The CIP frequency in ceramic UF was more than 30 days. Therefore, with actual conditions, ceramic UF membranes operation have a higher chemical consumption than polymeric UF.

4.1.2. Nanostructured Carbon performance

In **Figure *19*** is shown the evolution of flow and inlet pressure during CNM operation, the last shows that particular matter are retained into carbon particles, so the pressure is increased because there is a lower area to flow the water. In x-axis is shown the number of bed filtered, it is obtained dividing the volume filter by the column volume (0,25 m^3). Until now, the CNM has made near to 20.000 cycles and regeneration has not been needed.

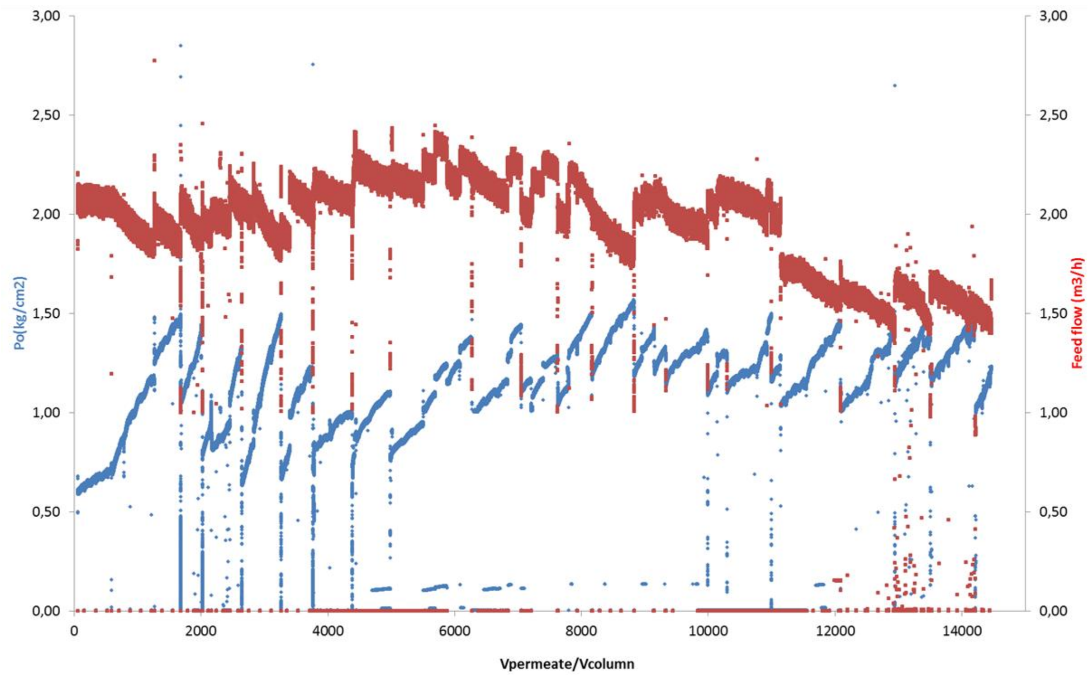

Figure 19: Evolution of inlet pressure and flow with volume filtrate

In **Figure *19*** can be seen that the slope of the inlet pressure is not constant, it is due to the quality of feed water. In **Table 11**, the different slopes are shown, can be seen that after the cycle 6 the slope is lower than in the first cycles. This difference is due to the chlorination in the tertiary treatment. During the first five cycles the tertiary was working without chlorination, after the six cycles the tertiary worked with chlorination and a concentration between 1-2 ppm of chlorine was detected in the inlet of the prototype. The average of the first five cycles was 0,024 bar/n; after the chlorination the slope was 0,010 bar/n. Therefore the chlorine reduces the effect of fouling to the half. This reduction may be due to the reduction of bacteria and other microorganism in the feed because of chlorine. Before chlorination there was microbiological growth, after this there was not any growth.

Table 11: Slopes for different cycles in CNM operation

Cycle	Slope·10^3 (bar/n)	Cycle	Slope·10^3 (bar/n)
1	12,90	10	10,59
2	25,85	11	13,58
3	22,22	12	4,67
4	30,12	13	8,70
5	23,58	14	9,07
6	6,62	15	9,22
7	10,53	16	11,57
8	10,40	17	8,65
9	13,61		

The operation conditions can be seen in **Table 12**. There are two operation conditions, with 2 m^3/h and 1,5 m^3/h. In all cases, water yield is higher than 99% because of a high amount of water can be filtered until the carbon particles are full of pollutants. The chemical consumption is zero in both cases due to chemicals are not needed for backwash. The BW frequency is 150 m^3 filtered for a feed flow of 2,0 m^3/h, and 269 m^3 for 1,5 m^3/h.

Table 12: Operating conditions of CNM with a feed flow of 2,00 m^3/h

Parameters	1^{st} conditions	2^{nd} conditions
Feed flow (m^3/h)	2,00-2,20	1,50-1,70
Filtration velocity (m/h)	10,2	7,6
BW frequency	Every 72 hours or 150 m^3	Every 168 hours or 269 m^3
Water yield (%)	99,8	99,9
Chemical consumption (mL/m^3)	0	0

The overall average of remove turbidity was 61$\pm$15%, 33$\pm$28 of COD, and 42$\pm$20% of TOC. Sixteen samples were analysed to obtain those results. During the first month of operation, the highest removal efficiency was obtained, nearly to 80% for DQO and TOC. Over time removal efficiency was reduced and stabilized between 25 and 40 %. Therefore, there is a reduction of removal efficiency in COD, TOC, Fe and Ni.

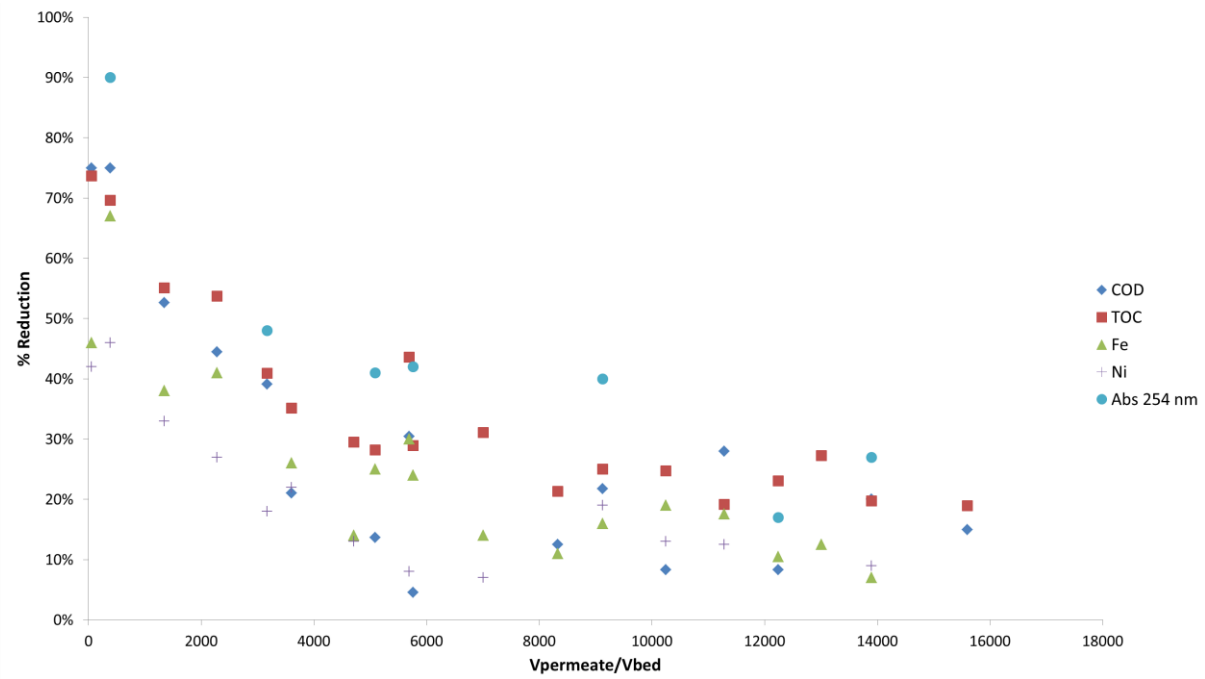

Figure 20: Evolution of CNM efficiency removal

In **Figure 21** and **Figure 22** can be seen the C/C_o curves for the pollutants assessed in CNM unit. From a theoretical point view, these curves should have "S" shape. The top of the curve indicate that the removal efficiency has been reduced and the carbon is near saturation, therefore is near to regeneration. Regeneration depends of removal efficiency, in this case is assessed the removal efficiency until it is zero. At the beginning of the operation, removal efficiency for COD and TOC is higher than limit of detection, therefore the real curves begins in the point $C/C_o = 0$.

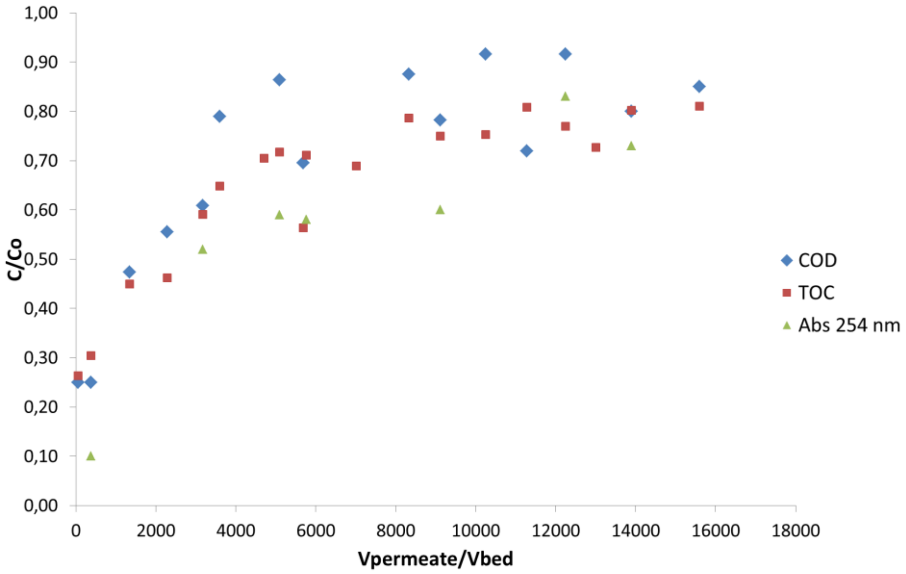

Figure 21: Curve C/Co for COD, Toc and absorbance at 254 nm

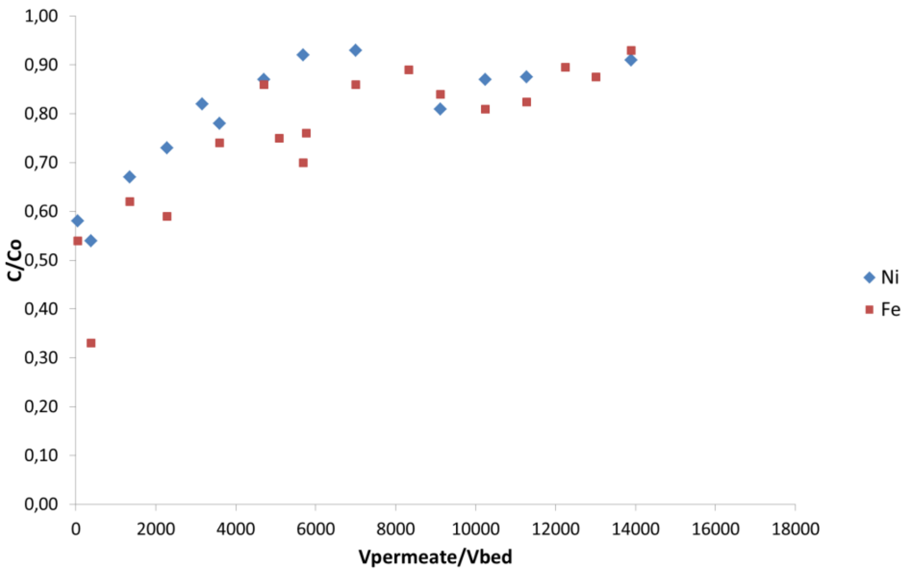

Figure 22: Curve C/Co for Fe and Ni

During operation time has been analysed different micropollutans, but not all of them were found because of limit of detection (LoD). The limit of detection for BETEX was 8,00 μ/L and for PCB was 0,01 ng/L, those components were in all the samples under LoD, therefore the removal efficiency of them could not be assessed. Not all the triazines, PAH and pesticides were found in detectable amounts, but some of them were. In **Table 13** are summarised the micropollutans found and their removal efficiency.

Table 13: CNM removal efficiency of micropollutants

	Average	St.dev
Atrazine	100%	0%
Sebutilazina	100%	0%
t-butilazina	39%	44%
Total Triazines	**49%**	**38%**
Acenaphthene	54%	41%
Anthracene	46%	40%
Benzo (a) anthracene	11%	3%
Benzo (a) pyrene	100%	0%
Benzo (a) fluoranthene	45%	50%
Benzo (a) pyrene	100%	0%
Benzo (k) fluoranthene	48%	48%
Fluorene	27%	20%
Naphthalene	45%	7%
Pyrene	40%	30%
Total PAH	**57%**	**50%**
a-Hexachlorocyclohexane	56%	9%
Aldrin	4%	0%
Alpha-endosulfan	53%	45%
b-hexachlorocyclohexane	33%	25%
Chlordane	100%	0%
d-Hexachlorocyclohexane	30%	40%
g-HCH (Lindane)	36%	19%
Heptachlor	31%	24%
Metoxichlor	100%	0%
p-p'-DDD	100%	0%
p-p'-DDE	6,%	4%
p-p'-DDT	9%	0%
Total Pesticides	**34%**	**23%**

To compare the results obtained in this work, a similar process with activated carbon was found. In Mailler et all (Mailler et all., 2016), has been studied a new tertiary treatment to remove micropollutants and organic matter from water. These new treatments use of a micro activated carbon with a bulk density of 0,53 kg/m^3, a median particle size of 423,5 μm and a specific BET surface of 860 m^2/g (Mailler et al. 2016).

Table 14: Comparison of % removal between work made in Paris by Mailler et al. and LIFE WIRE project.

Parameter	Activated carbon by Mailler et al.	CNM by LIFE WIRE
COD	21-48	75-8
TOC	13-44	74-18
Abs 254 nm	22-48	90-17

Table 14 shows higher removal efficiency for CNM than activated carbon. The removal efficiency for activated carbon is between 21 and 48 % for COD, 13-44 for TOC and 22-48 for Abs at 254 nm. With CNM the removal efficiency for COD ranged between 75% and 8%, 74-18 % for TOC and 90-17% for abs at 254 nm.

In the study made by Mailler et al.(Mailler et al.,2016) was assessed the relation between absorbance removal and TOC and COD removal efficiency. This is a useful aspect, because it allows monitoring of operation through the absorbance analysis.

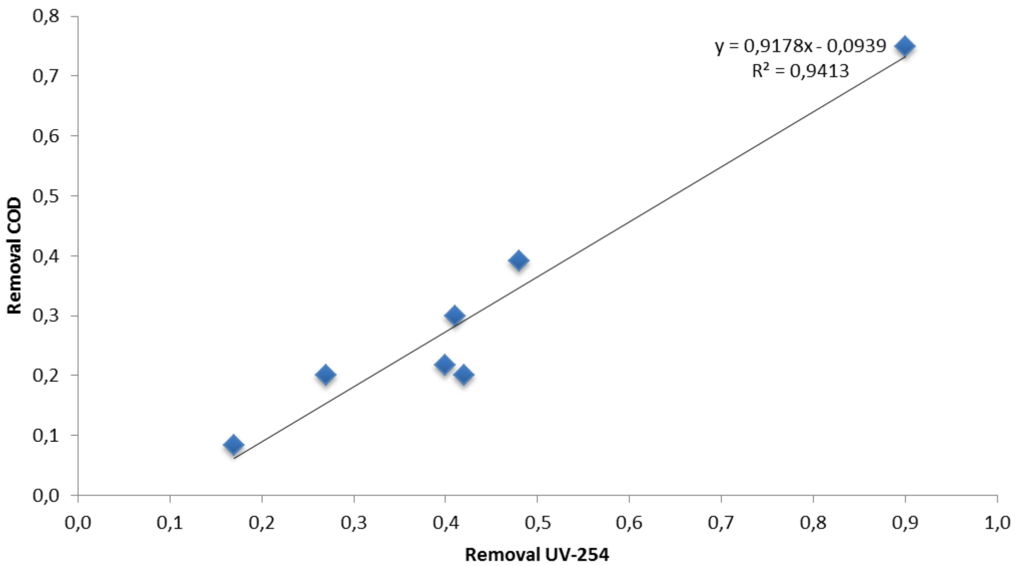

Figure 23: Correlation between UV-254 and COD removals for samples of the CNM pilot

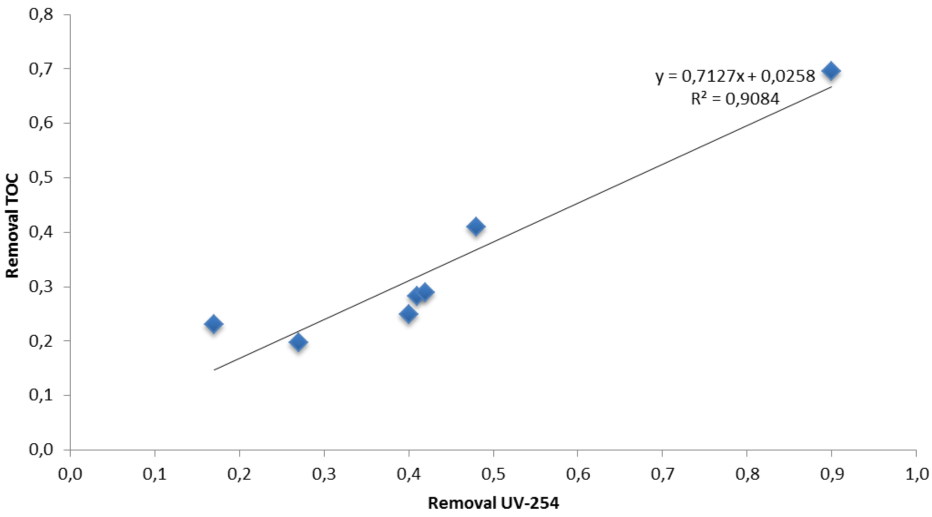

Figure 24: Correlation between UV-254 and TOC removals for samples of the CNM pilot

In **Figure 23** and **Figure 24** is shown the correlation between UV-254, COD and TOC removals. The correlation coefficient is 0,94 for COD and 0,9 for TOC. In both cases can be seen that the UV-254 can be an indicator of COD and TOC removal efficiency.

The removal of pesticides using activated carbon was >60%, higher than the removal efficiency obtained by CNM. The removal efficiency for triazines and PAH were not assessed with activated carbon, therefore cannot be compared.

4.1.3. Reverse osmosis performance

In **Table 15** are shown the RO conditions assessed, all the conditions tested were with an average recovery of 41±1 %. The first conditions tested were with a feed flow of 2 m^3/h, and were tested with and without flushing. Flushings increase the days before CIP from 17 days to 23 days, and not affect to the water yield because is only a flushing per day of 90 seconds with a flow of 2 m^3/h. Therefore operate with flushing is better than operate without flushing. The salt passage for the first conditions was 0,8 % for the first stage and 0,7% for the second stage. The feed pressure was between 10,5 and 11,0 bar, obtained a power consumption of 0,90 kW·h/m^3 of permeate. The consumption of Genesys RED was 3,31 mL/m^3 treated and 2,98 mL/m^3 for Genesys LF. The manufacturer establishes the dose of Genesys LF; the dose of Genesys RED depends of the chlorine content in feed water, and therefore cannot be adjusted.

The second conditions tested were with a feed flow of 1,50 m^3/h, the salt passage were 1,3 % for the first stage and 0,8 % for the second stage. In this conditions the feed pressure was between 7,0 and 7,5 bar, obtained and power consumption of 0,72 kW·h/m^3 permeate.

Table 15: RO operation conditions

Parameters	1st conditions	2nd conditions
Feed flow (m^3/h)	2,00-2,10	1,5
Recovery (%)	41±1	41±1
Permeate flow 1st /2nd stage (m^3/h)	0,58/0,26	0,44/0,22
Salt passage 1st/2nd stage (%)	0,8/0,7	1,3/0,8
Feed pressure (bar)	10,5-11,0	7,0-7,5
Pressure drop 1st stage (bar)	0,9	0,8-0,9
Power consumption (kW·h/m^3 perm)	0,90	0,72
CIP frequency No flushing 1 flushing/day	Every 17 days or 513 m^3 Every 23 days or 737 m^3	- Every 33 days or 1083 m^3
Chemical consumption		
Genesys RED (mL/m^3 treated)	3,3	5,3
Genesys LF (mL/m^3 treated)	3,0	2,2
NaOH (50%) (L/CIP)	0,5	0,5
HCl (15%) (L/CIP)	2,8	2,8

To study RO fouling and scaling, normalized permeate flow rate must be studied. The manufacturer of the RO membranes (Hydranautics) established that if a flow is reduced in a 15%, RO should be cleaned with chemicals to prevent membrane damage and irreversible fouling.

In **Figure 25** is shown the evolution of the permeate flow and between bars the different conditions tested. The left side of the **Figure 25** is during the start-up. Can be seen that the flow of the first stage is reduced harder than the second stage flow. It is because if some big particles enter in the RO system, those are fed in the first stage, so it will suffer more hardly conditions. The permeate flow of the second stage is constant and need lower CIP frequency.

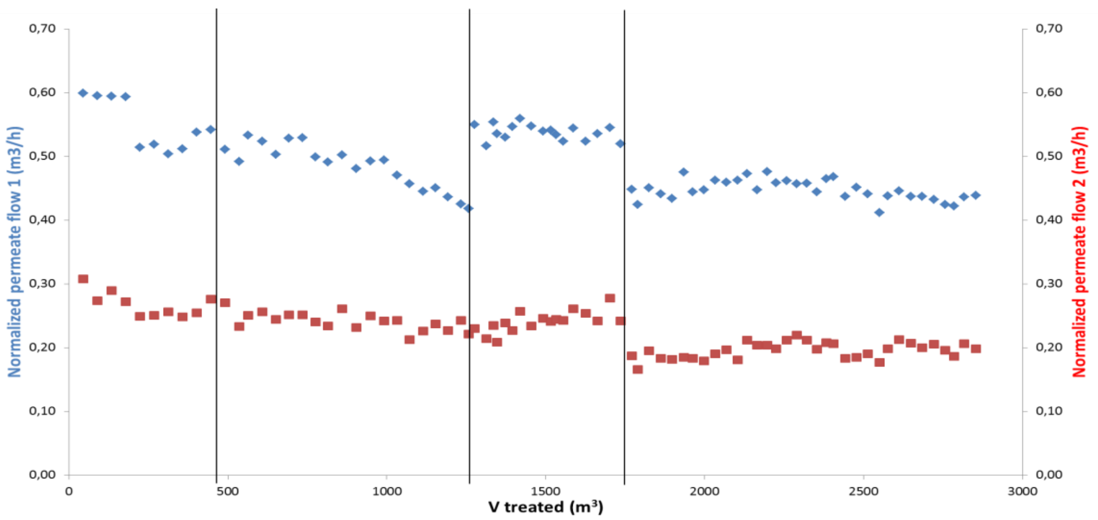

Figure 25: Evolution of permeate flow in RO as a function of the treated volume

To recover the membrane permeability and the membrane flux, a CIP is necessary when the conditions before mentioned happens. It is in two stages, the first stage is a basic cleaning (NaOH, 800 ppm), and the second stage is an acid clean (HCl, 1400 ppm). The protocol is the same for both stages. First of all, the membrane is cleaned with osmotised water during 10 minutes. After this, the chemical solution is recirculate during 1 hour (0,7 m^3/h for 10 minutes, 1,3 m^3/h for 10 minutes and 2,0 m^3/h for 40 minutes). This recirculation step is followed by a soaking stage during 30 minutes. The last step of the CIP is a flushing during 10 minutes to removes the chemicals from the RO unit. Between basic and acid stage, a flushing is made. In the experiments made by Ordoñez et all., RO operated between 15 and 25 days before CIP. In this study, RO achieved a CIP requirement after 23 days.

In **Table 16** are summarised the evolution of DQO, TOC and $N\text{-}NH_3$. Those parameters are tracked because can indicate if there are any problem in RO. It should be noted that the limit of detection (LoD) restricts the % of reduction. LoD to COD was 5 $mg{\cdot}L^{-1}$ until the week 8, after week 8 it was reduced to 4 $mg{\cdot}L^{-1}$ because water requirements are 4 $mg{\cdot}L^{-1}$. For TOC same situation was observed, until week 8 Lod was 2 $mg{\cdot}L^{-1}$, after week 8 it was reduced to 0,1 $mg{\cdot}L^{-1}$.

Table 16: Evolution of mean RO quality parameters

V treated (m^3)	Parameter					
	COD ($mg{\cdot}L^{-1}$)		TOC ($mg{\cdot}L^{-1}$)		N_2 ammonia ($mg{\cdot}L^{-1}$)	
	Inlet	Outlet	Inlet	Outlet	Inlet	Outlet
20	19	<5	6,9	<2	1,1	0,1
134	18	<5	6,8	<2	3,6	0,1
208	9	<5	3,1	<2	1,4	0,1
491	10	<5	3,7	<2	2,8	0,1
608	14	<5	6,5	<2	1,1	0,1
815	15	<5	4,8	<2	3,4	0,1
991	24	<5	5,5	<2	1,5	0,1
1193	16	<4	4,4	<0,2	0,9	0,1
1258	21	<4	5,9	0,8	2,9	0,1
1347	26	6	6,2	<0,5	2,4	0,1
1419	21	<4	7,0	<0,5	2,2	0,1
1516	18	<4	7,2	<0,5	0,8	0,1
1588	22	<4	7,3	<0,5	1,3	0,1
1702	18	<4	7,6	<0,5	1,3	0,1
1898	25	<4	6,4	<0,5	0,3	0,1
2166	16	<4	6,5	<0,5	0,3	0,1
2405	17	<4	6,0	<0,5	1,2	0,1
2853	18	<4	6,3	<0,5	1,5	0,1

In **Figure 26** can be seen the evolution of the percentage of reduction as a function of the treated volume. But must be observed with table 10 because a lower % reduction can be due to a low inlet concentration and it is limited by the LoD. The reduction of COD is always under the LoD, except in one sample, it can be due to problems in analysis or when the samples were taken, because it is a very high concentration for RO permeates. For TOC the outlet concentration is under LoD, after week 8 it is near to 0,5 mg·L^{-1} that means a percentage of reduction near to 90%. This shows high removal efficiency as N-NH_3, which were below the limit of detection.

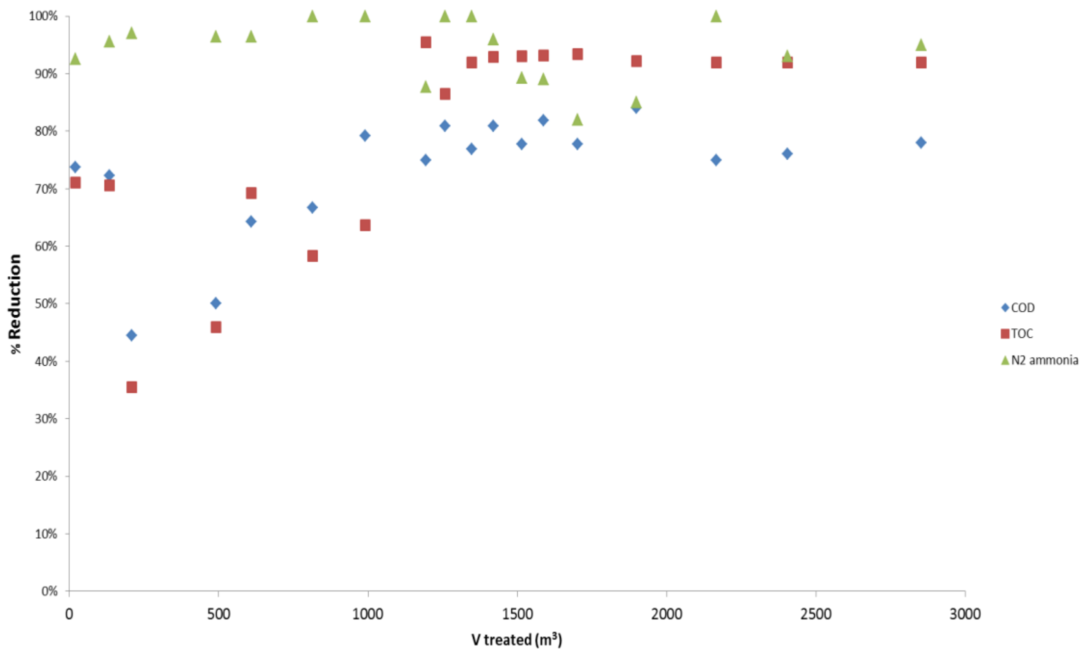

Figure 26: Evolution of RO efficiency removal as a function of treated volume

4.1.4. Results of Configuration I

In **Table 17** is shown the water quality obtained by UF, CNM and CNM-RO. UF unit removes mainly phosphates, oily matter and bacteria content, and those are expected results because of the pore diameter of UF membrane. CNM unit removes COD because of removes compounds with carbon content. To remove other species the RO unit is needed

Table 17: Water quality in Configuration I

Parameter	Raw Water	UF permeate	CNM permeate	RO permeate
Iron (mg·L^{-1})	0,041±0,091	0,03±0,007	0,03±0,02	<0,006
Copper (mg·L^{-1})	<0,002	<0,002	<0,002	<0,002
Total hardness (ºF)	48,3±3,5	45,7±3,4	47,7±2,97	0,10±0,03
pH	7,74±0,17	7,69±0,14	7,51±0,16	5,75±0,19
Oily matter (mg·L^{-1})	0,82±0,53	<0,5	<0,5	<0,5
Alkalinity (meq·L^{-1})	2,85±0,23	2,59±0,25	2,66±0,22	0,05±0,01
SiO_2 (mg·L^{-1})	9,49±0,94	9,37±1,22	9,58±1,22	<0,02
Phosphates (mg·L^{-1})	5,46±1,90	4,32±2,00	5,19±2,16	0,12±0,06
Suspended solids (mg·L^{-1})	<3	<3	<3	<3

Conductivity	2103±111	2040±91	2094±98	24,58±6,92
Chloride (mg·L^{-1})	375±75	356±97	388±54	4,77±1,90
COD (mg·L^{-1})	22,00±3,46	19,4±2,91	16,86±4,07	<4
Bacteria (CFU·mL^{-1})	100,3±44,1	70,37±55,20	969±1357	19,1±2,4

In **Figure 27** can be seen the evolution of the target analysed species through the different units. All the parameters are mainly remove in the RO unit; therefore the RO is the most important unit from the point of view of water quality. The CNM and UF remove partly some compounds; the RO pre-treatment helps the RO operation and reduces the time until CIP.

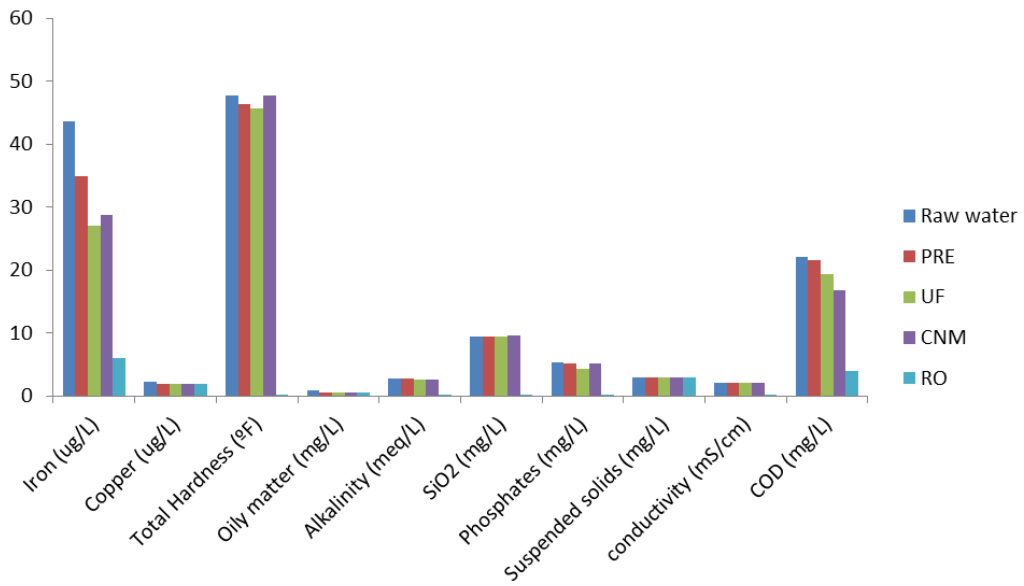

Figure 27: Evolution of the target analysed species in the different units

In **Table *18*** can be seen that the combination of CNM and RO meet the requirements to boiler and cooling applications. CNM a UF cannot meet requirements due to the conductivity and hardness requirements.

Table 18: Comparison between water requirements and water obtained in the different units

Use	Raw Water	UF permeate	CNM permeate	RO permeate
Boiler water	X	X	X	√
Cooling water	X	X	X	√

In **Table 19** is summarised the operation of configuration I. The system CNM+RO obtain a total water yield of 41%. The power consumption is 0,95 kW·h/m^3 permeate, it is lower than the energy consumption of a UF-RO system, and the chemical consumption is higher for UF-RO systems due to UF chemical consumption. CNM water yield is higher than UF water yield because of CNM can work during 3 days continuously and UF operate in smaller cycles than CNM. The overall water yield to a CNM-RO system is 40%.

The overall chemical consumption for CNM-RO system is due to the RO unit. The consumptions are given by m^3 fed, the operation conditions were 2,0 m^3/h, one flushing per day and CIP requirements 23 days. The consumption of Genesys RED and Genesys LF are 3,3 a 3,0 mL/m^3 respectively. NaOH and HCl are used during CIP, but those consumptions are given for m^3 fed to RO, the calculations were made by the volume treated until CIP. The consumption of NaOH (50%) is 0,4 mL/m^3, and to HCl (15%) is 0,2 mL/m^3.

UF unit operates with a feed flow of 1,00 m^3/h (45 Lmh) and without reject current. Due to the low efficiency of BW, this were remove of the process and one basic CEB is made each 6 hours, without acid CEB. In this conditions the CIP frequency is more than 30 days.

Table 19: Summary of flows and consumptions in configuration I

Parameter	PRE	UF	CNM	RO
Feed flow (m^3/h)	4,00	1,00	2,10	2,05
Permeate flow (m^3/h)	4,00	1,00	2,10	0,84
Rejected flow (m^3/h)	-	-	-	1,21
Water yield (%)	98,9	96,4	99,8	41,0
BW frequency	1 day	-	3 days	1 days
CIP frequency	-	15 days	-	23 days
Power consumption ($kW·h/m^3$)		0,045	0,036	0,905
Chemical consumption (mL/m^3)				
HClO (15%)	-	1560	-	-
HNO_3 (65%)	-	-	-	-
$FeCl_3$ (40%)	-	20	-	-
NaOH (50%)	-	250	-	0,4
HCl (15%)	-	-	-	0,2
Genesys RED	-	-	-	3,3
Genesys LF	-	-		3,0

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Conclusions

From a hydraulic point of view, all the units except UF were working continuously during 8 months treating the reclaimed water from El Baix Llobregat WWRP. The CNM unit obtained a water yield higher than 99%, without chemical consumption. After 8 months of operation no regeneration or chemical treatment has been made.

The combination of CNM-RO worked continuously, with a CIP requirements for RO unit of 23 days or 737 m^3. This CIP frequency means that the chemical consumptions is low and the RO unit can work more days continuously. The chemical and electrical consumptions are lower than advanced treatment in El Baix Llobregat WWRP, which use polymeric UF+RO. Therefore, the CNM-RO treatment presents a real alternative to a UF+RO conventional treatment.

All the experiments in UF unit were made with 44,8 LMH or more. Therefore the ceramic UF can not operate without CEB at flux higher than 44,8 LMH with reclaimed water. Operating with one basic CEB each 8 hours and one acid CEB each 3 days allows a continuously operation, with CIP requirements of more than 30 days.

From a quality perspective, all the requirements were achieved with the CNM+RO scheme. The requirements were met during the 8 months of operation; therefore this scheme can be implemented in a company to guaranty a water quality, despite of the variations in reclaimed water. The UF and CNM cannot be used to those applications, due to the hardness and salt requirements.

In parallel, the CNM was assessed from the point of view of micropollutants. The removal efficiency obtained was $49\pm38\%$ for triazines, $57\pm50\%$ for PAH and $34\pm22\%$ for pesticides. These results demonstrate a real alternative in advanced wastewater treatments to remove micropollutants, meanly triazines, PAH and pesticides.

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Máster Universitario en Ingeniería Química

Junio 2016

Treball de Fi de Máster

Máster Universitario en Ingeniería Química

Tertiary effluent treatment using membranes and adsorption technologies for industrial reuse

ANNEX A: PID of prototype

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Director/s: Jose Luis Cortina Pallas
Ponent: Sandra Casas Garriga
Convocatòria: Junio 2016

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d'Enginyeria Industrial de Barcelona

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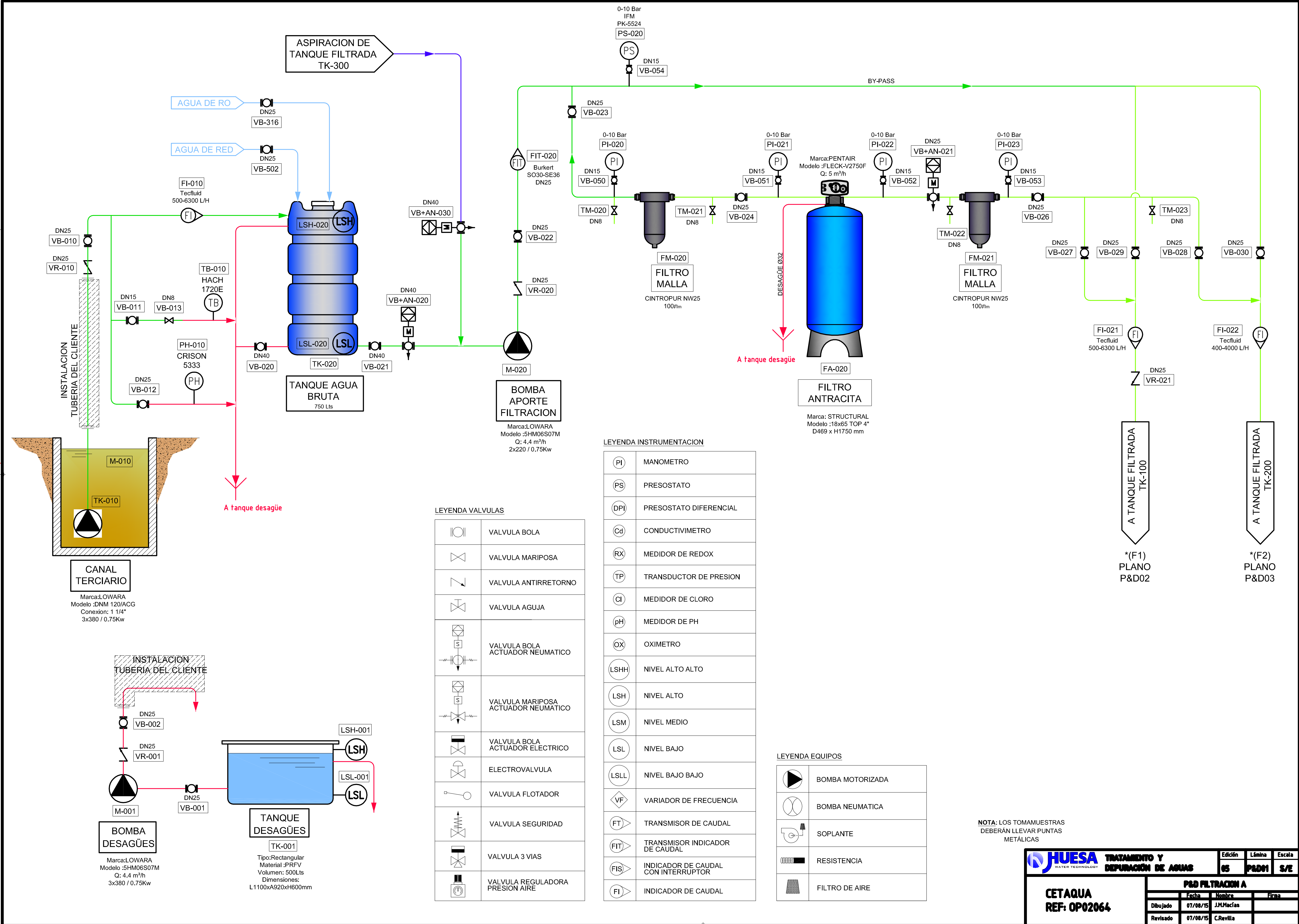

LEYENDA VALVULAS

	VALVULA BOLA
	VALVULA MARIPOSA
	VALVULA ANTIRRETORNO
	VALVULA AGUJA
	VALVULA BOLA ACTUADOR NEUMATICO
	VALVULA MARIPOSA ACTUADOR NEUMATICO
	VALVULA BOLA ACTUADOR ELECTRICO
	ELECTROVALVULA
	VALVULA FLOTADOR
	VALVULA SEGURIDAD
	VALVULA 3 VIAS
	VALVULA REGULADORA PRESION AIRE

LEYENDA INSTRUMENTACION

PI	MANOMETRO
PS	PRESOSTATO
DPI	PRESOSTATO DIFERENCIAL
Cd	CONDUCTIVIMETRO
RX	MEDIDOR DE REDOX
TP	TRANSDUCTOR DE PRESION
Cl	MEDIDOR DE CLORO
pH	MEDIDOR DE PH
OX	OXIMETRO
LSHH	NIVEL ALTO ALTO
LSH	NIVEL ALTO
LSM	NIVEL MEDIO
LSL	NIVEL BAJO
LSLL	NIVEL BAJO BAJO
VF	VARIADOR DE FRECUENCIA
FT	TRANSMISOR DE CAUDAL
FIT	TRANSMISOR INDICADOR DE CAUDAL
FIS	INDICADOR DE CAUDAL CON INTERRUPTOR
FI	INDICADOR DE CAUDAL

LEYENDA EQUIPOS

	BOMBA MOTORIZADA
	BOMBA NEUMATICA
	SOPLANTE
	RESISTENCIA
	FILTRO DE AIRE

NOTA: LOS TOMAMUESTRAS DEBERÁN LLEVAR PUNTAS METÁLICAS

HUESA WATER TECHNOLOGY	TRATAMIENTO Y DEPURACIÓN DE AGUAS	Edición	Lámina	Escala
		05	P&D01	S/E
CETAQUA REF: OP02064	P&D FILTRACION A			
		Fecha	Nombre	Firma
	Dibujado	07/08/15	J.M.Macías	
	Revisado	07/08/15	C.Revilla	

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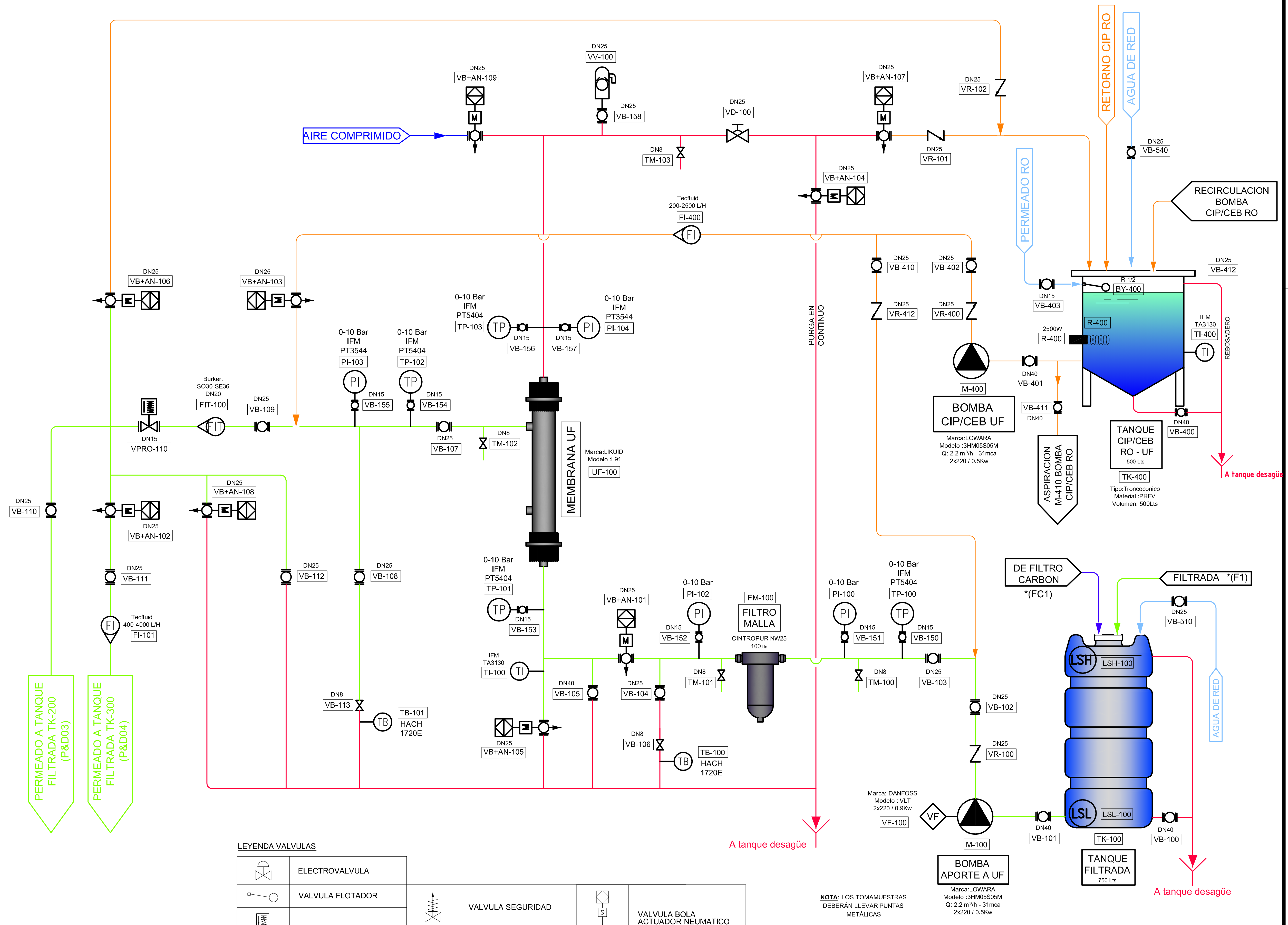

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VALVULA PROPORCIONAL	VALVULA MARIPOSA
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		005	P&D02	S/E

CETAQUA REF: OP02064	P&D UF			
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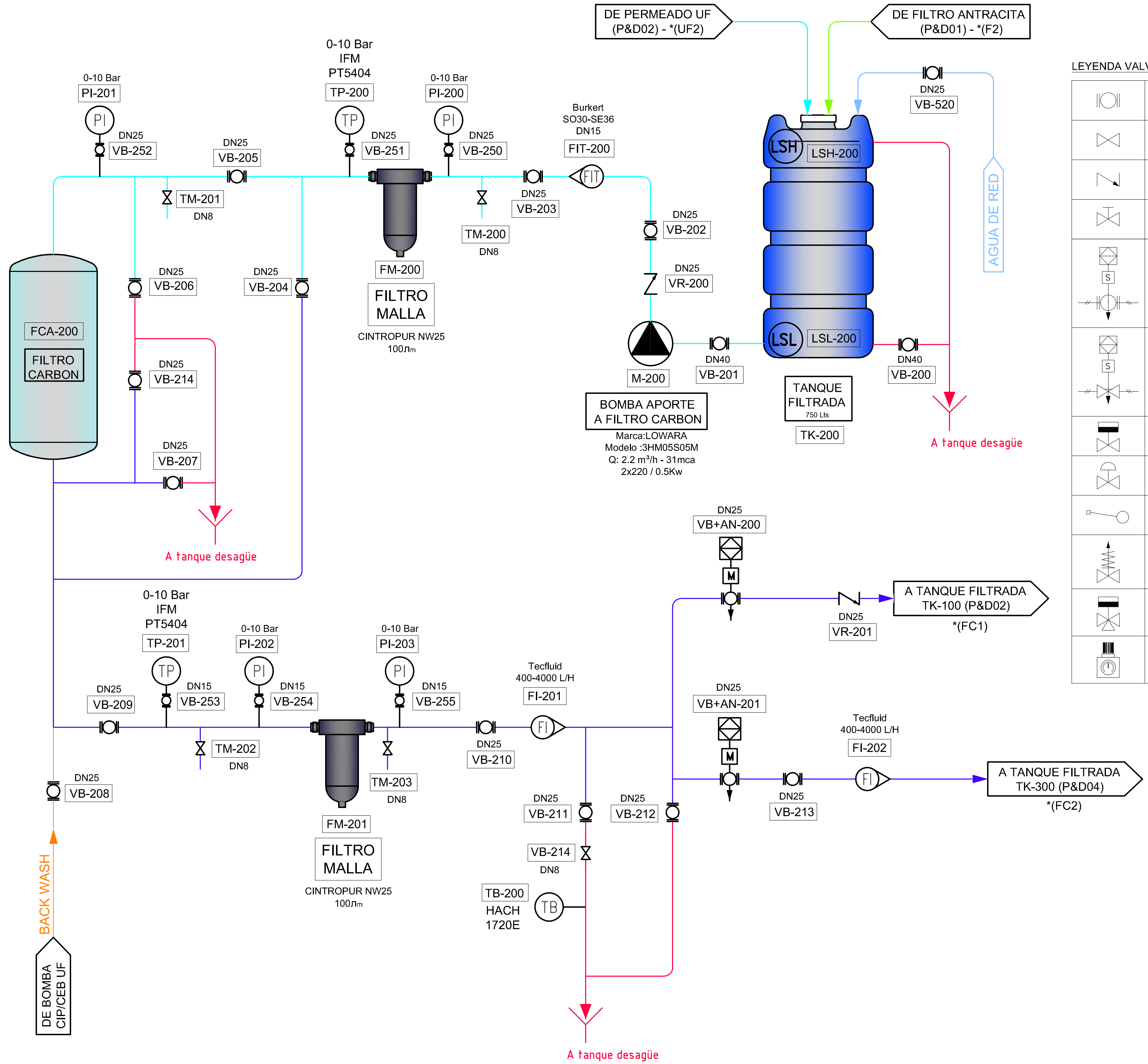
	Revisado	07/08/15	Martín L.B.	



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pH	MEDIDOR DE PH
OX	OXIMETRO
LSHH	NIVEL ALTO ALTO
LSH	NIVEL ALTO
LSM	NIVEL MEDIO
LSL	NIVEL BAJO
LSLL	NIVEL BAJO BAJO
VF	VARIADOR DE FRECUENCIA
FT	TRANSMISOR DE CAUDAL
FIT	TRANSMISOR INDICADOR DE CAUDAL
FIS	INDICADOR DE CAUDAL CON INTERRUPTOR
FI	INDICADOR DE CAUDAL

LEYENDA EQUIPOS

	BOMBA MOTORIZADA
	BOMBA NEUMATICA
	SOPLANTE
	RESISTENCIA
	FILTRO DE AIRE

NOTA: LOS TOMAMUESTRAS DEBERÁN LLEVAR PUNTAS METÁLICAS

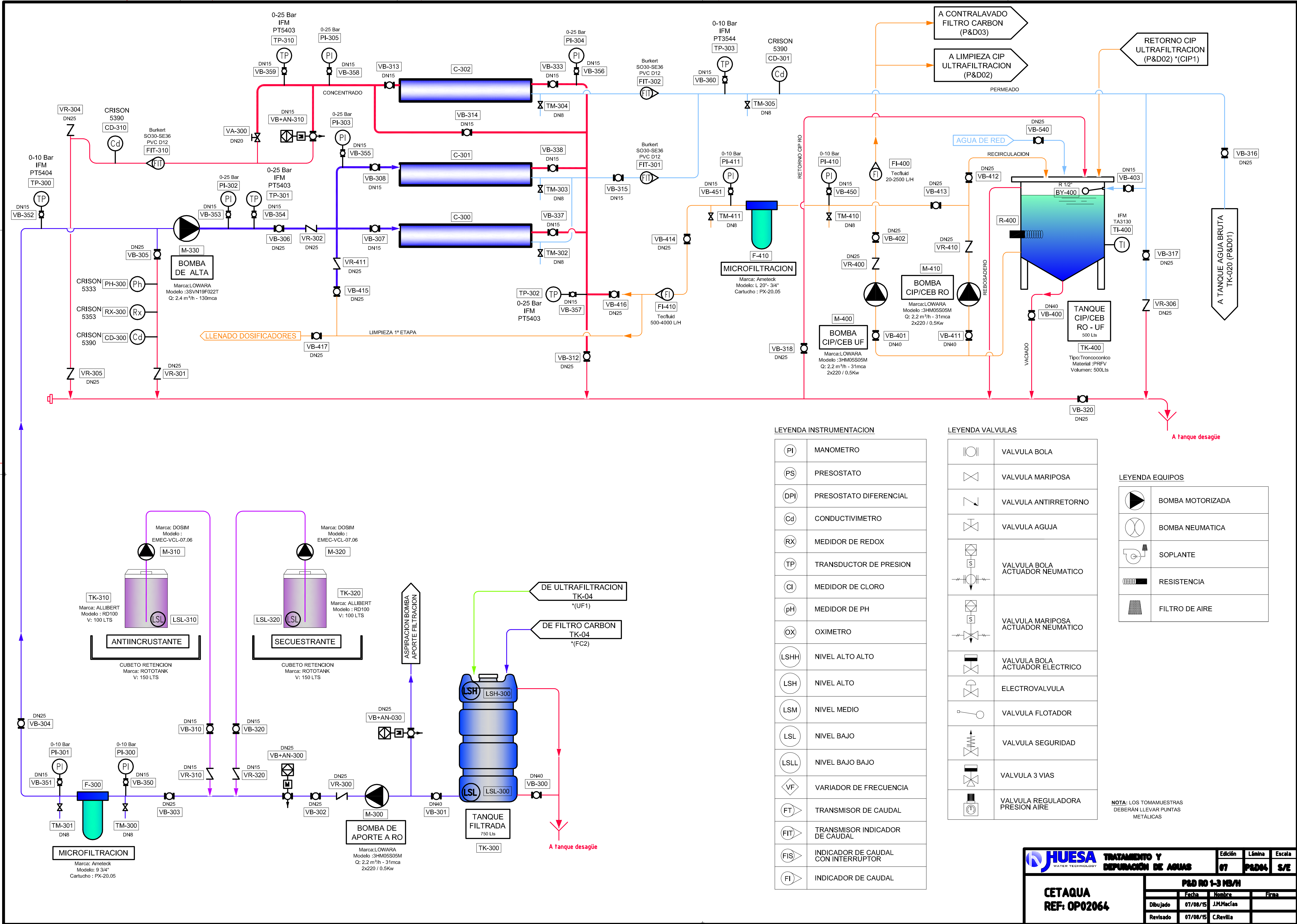
HUESA WATER TECHNOLOGY — TRATAMIENTO Y DEPURACIÓN DE AGUAS

Edición	Lámina	Escala
05	P&D03	S/E

P&D FILTRACION CARBON

CETAQUA
REF: OP02064

	Fecha	Nombre	Firma
Dibujado	07/08/15	J.M.Macias	
Revisado	07/08/15	C.Revilla	



A CONTRALAVADO FILTRO CARBON (P&D03)
A LIMPIEZA CIP ULTRAFILTRACION (P&D02)
RETORNO CIP ULTRAFILTRACION (P&D02) *(CIP1)
A TANQUE AGUA BRUTA TK-020 (P&D01)
PERMEADO
CONCENTRADO
RECIRCULACION
RETORNO CIP RO
REBOSADERO
VACIADO
AGUA DE RED
LLENADO DOSIFICADORES
LIMPIEZA 1ª ETAPA
A tanque desagüe
C-300
C-301
C-302
BOMBA DE ALTA
M-330
Marca:LOWARA
Modelo :3SVN19F022T
Q: 2,4 m³/h - 130mca
CRISON 5333 PH-300
CRISON 5353 RX-300
CRISON 5390 CD-300
CRISON 5390 CD-310
CRISON 5390 CD-301
0-25 Bar IFM PT5403 TP-310
0-25 Bar IFM PT5403 TP-301
0-25 Bar IFM PT5403 TP-302
0-10 Bar IFM PT5404 TP-300
0-10 Bar IFM PT3544 TP-303
Burkert SO30-SE36 PVC D12 FIT-310
Burkert SO30-SE36 PVC D12 FIT-302
Burkert SO30-SE36 PVC D12 FIT-301
FI-410 Tecfluid 500-4000 L/H
FI-400 Tecfluid 20-2500 L/H
MICROFILTRACION F-410
Marca: Ameteck
Modelo: L 20"- 3/4"
Cartucho : PX-20.05
BOMBA CIP/CEB RO M-410
BOMBA CIP/CEB UF M-400
Marca:LOWARA
Modelo :3HM05S05M
Q: 2,2 m³/h - 31mca
2x220 / 0,5Kw
TANQUE CIP/CEB RO - UF 500 Lts TK-400
Tipo:Troncoconico
Material :PRFV
Volumen: 500Lts
R-400
BY-400
R 1/2"
IFM TA3130 TI-400
Marca: DOSIM
Modelo : EMEC-VCL-07.06
M-310
M-320
TK-310
TK-320
Marca: ALLIBERT
Modelo : RD100
V: 100 LTS
LSL-310
LSL-320
ANTIINCRUSTANTE
SECUESTRANTE
CUBETO RETENCION
Marca: ROTOTANK
V: 150 LTS
ASPIRACION BOMBA APORTE FILTRACION
DE ULTRAFILTRACION TK-04 *(UF1)
DE FILTRO CARBON TK-04 *(FC2)
TANQUE FILTRADA 750 Lts TK-300
LSH-300
LSL-300
BOMBA DE APORTE A RO M-300
MICROFILTRACION F-300
Marca: Ameteck
Modelo: 9 3/4"
Cartucho : PX-20.05
LEYENDA INSTRUMENTACION
PI MANOMETRO
PS PRESOSTATO
DPI PRESOSTATO DIFERENCIAL
Cd CONDUCTIVIMETRO
RX MEDIDOR DE REDOX
TP TRANSDUCTOR DE PRESION
Cl MEDIDOR DE CLORO
pH MEDIDOR DE PH
OX OXIMETRO
LSHH NIVEL ALTO ALTO
LSH NIVEL ALTO
LSM NIVEL MEDIO
LSL NIVEL BAJO
LSLL NIVEL BAJO BAJO
VF VARIADOR DE FRECUENCIA
FT TRANSMISOR DE CAUDAL
FIT TRANSMISOR INDICADOR DE CAUDAL
FIS INDICADOR DE CAUDAL CON INTERRUPTOR
FI INDICADOR DE CAUDAL
LEYENDA VALVULAS
VALVULA BOLA
VALVULA MARIPOSA
VALVULA ANTIRRETORNO
VALVULA AGUJA
VALVULA BOLA ACTUADOR NEUMATICO
VALVULA MARIPOSA ACTUADOR NEUMATICO
VALVULA BOLA ACTUADOR ELECTRICO
ELECTROVALVULA
VALVULA FLOTADOR
VALVULA SEGURIDAD
VALVULA 3 VIAS
VALVULA REGULADORA PRESION AIRE
LEYENDA EQUIPOS
BOMBA MOTORIZADA
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Edición 07
Lámina P&D04
Escala S/E
P&D RO 1-3 M3/H
CETAQUA
REF: 0P02064
Fecha Nombre Firma
Dibujado 07/08/15 J.M.Macías
Revisado 07/08/15 C.Revilla

Etiquetes dels CD's

Treball de Fi de Máster **Máster en Ingeniería Química**

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Ponent:
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Convocatòria:
Junio 2016

Tertiary efluent treatment using membranes and adsorption technologies for industrial reuse

Escola Tècnica Superior
d'Enginyeria Industrial
de Barcelona